

ARTISAN®
TECHNOLOGY GROUP

Your **definitive** source for quality pre-owned equipment.

Artisan Technology Group
(217) 352-9330 | sales@artisantg.com | artisantg.com

Full-service, independent repair center
with experienced engineers and technicians on staff.

We buy your excess, underutilized, and idle equipment
along with credit for buybacks and trade-ins.

Custom engineering
so your equipment works exactly as you specify.

- Critical and expedited services
- In stock / Ready-to-ship
- Leasing / Rentals / Demos
- ITAR-certified secure asset solutions

Expert team | Trust guarantee | 100% satisfaction

All trademarks, brand names, and brands appearing herein are the property of their respective owners.

Visit our website - *Click HERE*

OPERATING MANUAL FOR THE CRYOSTREAM COOLER

OCTOBER 1992

VERSION 3.1

OXFORD CRYOSYSTEMS

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

CONTENTS

SETTING UP

1) PRINCIPLE OF OPERATION
2) ITEMS REQUIRED
   - **2.1** COLDHEAD AND 611 CONTROLLER COMBINATION
   - **2.2** OPERATING VOLTAGE
   - **2.3** MOUNTING
   - **2.4** CONNECTING UP
3) EVAPORATOR

OPERATION

4) CPC611 TEMPERATURE CONTROLLER
   - **4.1** INSTRUCTIONS FOR USE
5) TROUBLESHOOTING

APPENDICES

- **I.** THE "ICING" PROBLEM
- **II.** DRY AIR SHROUD TUBE
- **III.** HOW CAN YOU KNOW THE TRUE TEMPERATURE AT THE CRYSTAL?
- **IV.** REMOTE INTERFACE
- **V.** CHART OUTPUT
- **VI.** PUMPING DOWN
- **VII.** THE DIAPHRAGM PUMP
- **VIII.** ICE BLOCKAGE
- **IX.** CRYOSTREAM SERVICE
- **X.** EVAPORATOR
  - AUTO MODE
  - MANUAL MODE

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

SETTING UP

1) PRINCIPLE OF OPERATION

The **CRYOSTREAM** is a new device used to cool small crystal specimens mounted on a diffractometer, in an X-ray camera, etc., and has been the subject of a publication (J.Cosier, A.M.Glazer, *J. Appl. Cryst.*, 19, 105-107 (1986)). Like many other liquid nitrogen devices, **CRYOSTREAM** produces a cold jet of gas that can be aimed at the specimen, but it is ***much more*** stable and is capable of refilling without any disturbance to the crystal temperature. This is achieved by separating the gas outflow from the liquid nitrogen supply vessel by a pump (*Figure 1*).

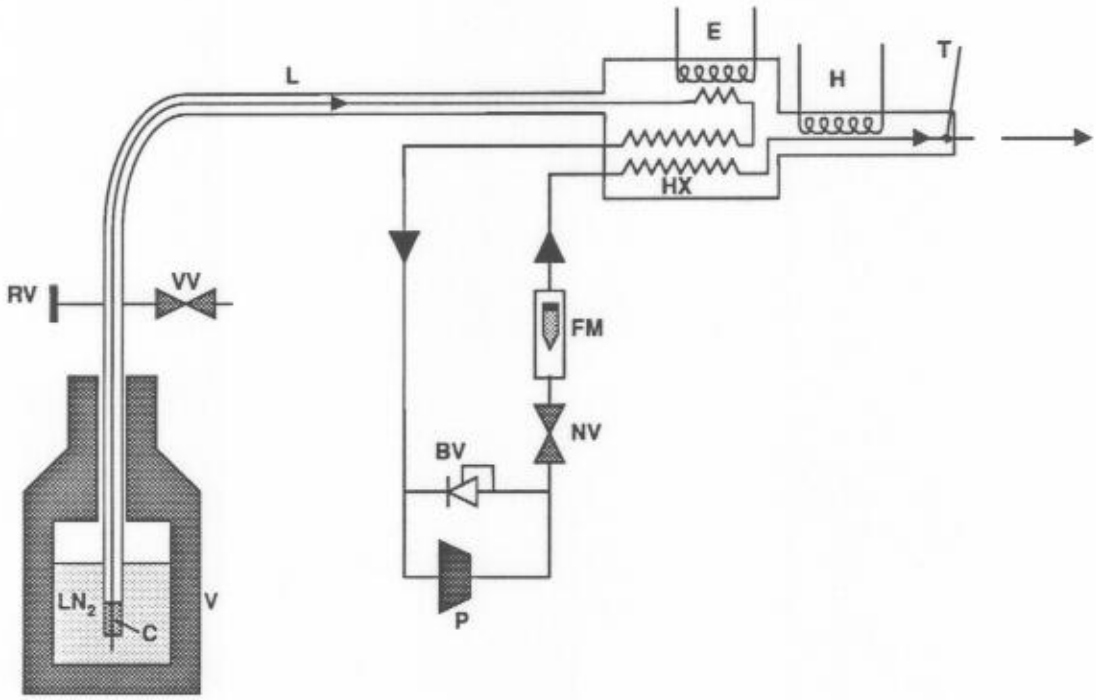


Figure 1 **CRYOSTREAM** Flow Scheme

Liquid nitrogen is drawn up by the action of the **diaphragm pump** (**P**) from an unpressurised **storage vessel** (**V**) through a flexible vacuum-insulated **supply line** (**L**). The storage vessel can be any convenient container of liquid nitrogen such as a conventional metal Dewar (**NOTE** that this is *unsealed*). The liquid nitrogen first passes through an electrically heated **evaporator coil** (**E**), which converts most of the liquid into vapour at the boiling point. This vapour flows outward along one path of the **heat exchanger** (**HX**) to arrive at the inlet of the diaphragm pump at

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

approximately 10K below room temperature. Gas from the pump flows inward along the other path of the heat-exchanger and is then recooled. Therefore, the main **heat-exchanger** (**HX**) carries the gas streams to and from the constant-flow **pump** (**P**), which operates at room temperature. The nitrogen flow rate is preset by a **needle valve** (**NV**). After returning to the cold end of the heat-exchanger, the gas enters the delivery nozzle to pass over a **heating coil** (**H**) and a **thermal sensor** (**T**) before emerging to cool the sample.

The flexible liquid-nitrogen supply line, the heat-exchanger, evaporator and delivery nozzle share a common high-vacuum insulation jacket which is pumped out and sealed before use. An adsorption **cryopump** (**C**) (charcoal) is build into the rigid part (leg) of the supply line. The temperature of the emerging gas stream is controlled by adjusting the **heater** (**H**) power in response to the temperature sensed by the **thermosensor** (**T**). The three-term programmable **CPC611** controller supplied by **Oxford Cryosystems** is used to control the temperature. In normal use, the **CRYOSTREAM** is capable of going from 80 to 375K with a stability of about 0.1K (temperatures as low as 77.4K have been achieved). *Figure 2* shows a plot of the variation in temperature over a 12 hour period when running at 100K.

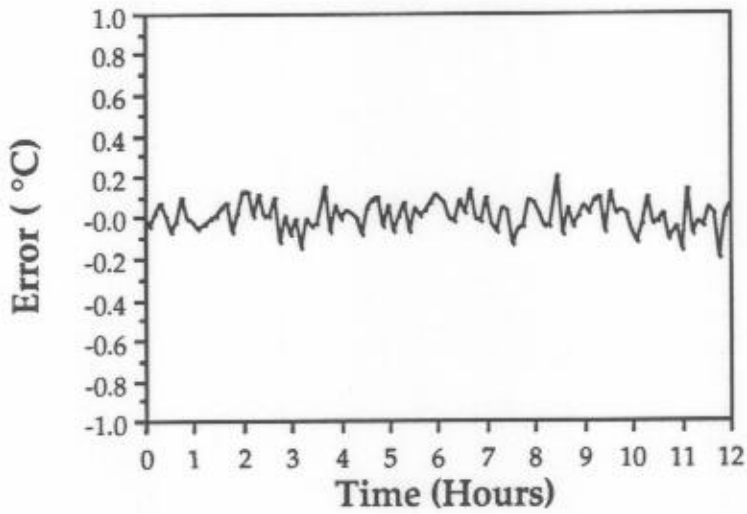


Figure 2 **CRYOSTREAM** Temperature Fluctuation at 100K

Because the flow rate is fixed by the needle valve at approximately 0.6 litres / hour of *liquid*, a 25 litre Dewar will last for more than one day of

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

continuous use. This usage does not depend on the temperature of the experiment. The supply vessel can be refilled at any time simply by pouring in liquid nitrogen.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

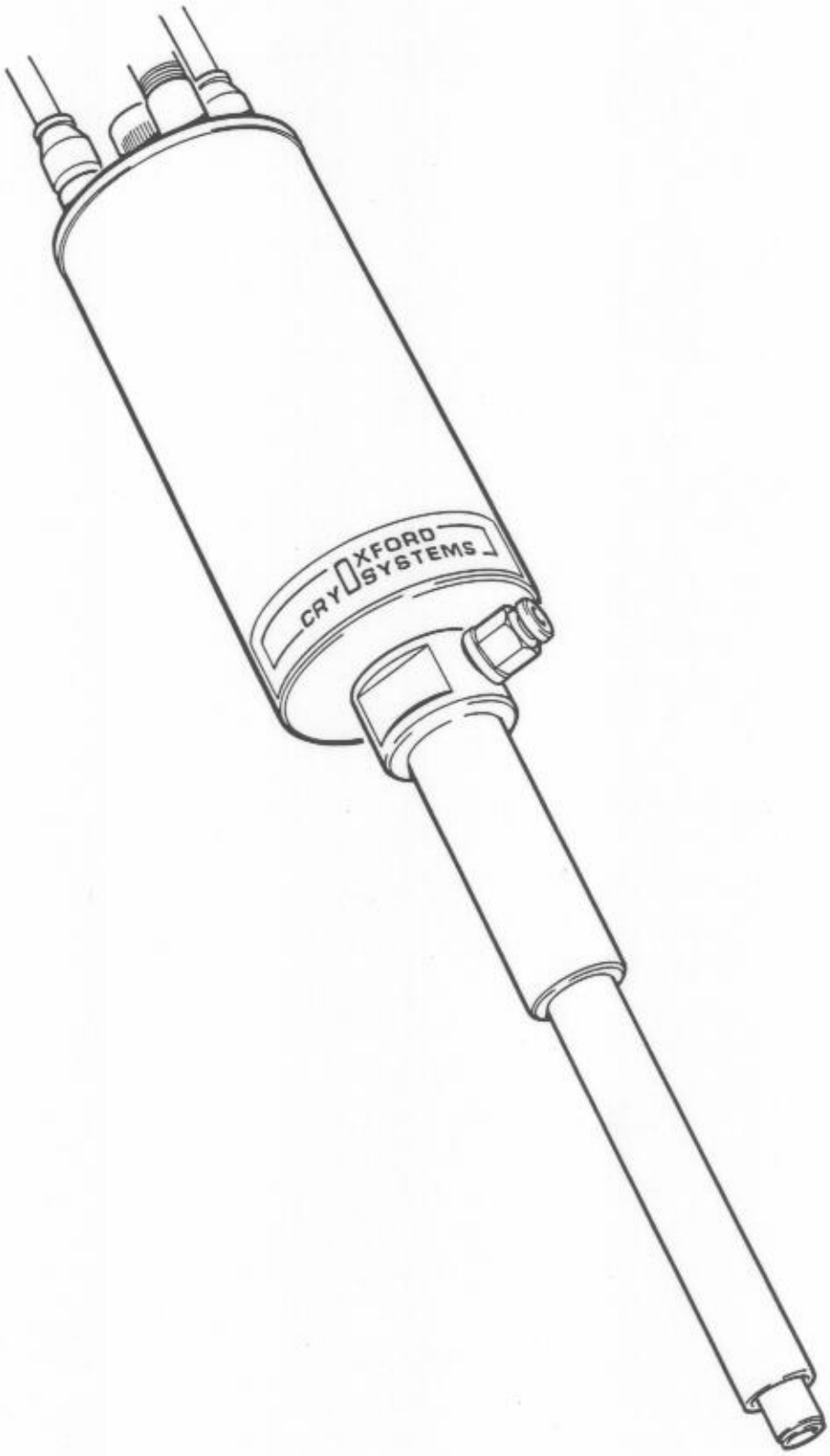


Figure 3 **CRYOSTREAM** Coldhead

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

2) ITEMS REQUIRED

Assembly of the **CRYOSTREAM** is simple as all connections are clearly marked. The component parts of the system are:
The **CRYOSTREAM** coldhead (*Figure 3*) - supplied
A diaphragm pump - supplied
A **CPC611** temperature controller (*Figure 5*)- supplied
A **CRH25** reactivation heater - supplied
An adaptor for diffractometer mounting - optional extra
A dry air unit - optional extra recommended for frost prevention

2.1 COLDHEAD AND 611 CONTROLLER COMBINATION

The **CRYOSTREAM** coldhead and 611 Controller are supplied as a matched pair - the individual characteristics of the coldhead sensors are programmed into the controller. ***Consult your supplier before attempting to operate unmatched units together.***

The serial number of the coldhead is engraved on the stainless steel top flange near to the flexible nitrogen line.
The serial number of the controller is shown on the top righthand corner of the rear panel and the number of the matched coldhead is shown near the coldhead connections on the rear panel.

2.2 OPERATING VOLTAGE

It is essential that **CRYOSTREAM** is configured to operate on the local mains electrical supply. Check the following:-

611 CRYOSTREAM Controller

The voltage selector switch on the rear panel must be set to the correct position, either
200-240 Volts A.C, 50Hz, 3 Amps
or
100-120 Volts A.C, 50-60Hz, 6 Amps

Make sure an 'Anti-Surge' (T) type fuse of the correct rating is fitted.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Gas Pump

Three voltage variants are currently available

220-240 Volts A.C, 50 Hz
115 Volts A.C, 60Hz
100 Volts A.C, 50-60Hz

Make sure you have the correct pump.

CRH25 Reactivation Heater

Two voltage variants are available - 230 Volts A.C and 115 Volts A.C

2.3 MOUNTING

Mounting of the **CRYOSTREAM** depends on the particular application. Oxford Cryosystems can supply suitable stands with which to mount **CRYOSTREAM** on a normal four-circle diffractometer (e.g. Stoe STADI4, Nicolet...) or on a Kappa geometry diffractometer (e.g. CAD4, Kuma KM4). Otherwise, any clamping arrangement will suffice (we leave this to the user!). It is perfectly safe to clamp the **CRYOSTREAM** by the 26mm diameter reinforced section of the nozzle (close to the **CRYOSTREAM** body). It is a good idea not to allow the **CRYOSTREAM** nozzle to point upward, otherwise there may be a problem with liquid nitrogen collecting inside. A typical arrangement that is commonly used on 4-circle diffractometers is shown in Figure 4, where the **CRYOSTREAM** is inclined over the top of the x-ray tube housing to point at the crystal. Typically the crystal should be less than 10 mm from the end of the nozzle.

NOTE: The supply line is made out of flexible tubing with a continuous metal inner tube. This should not be flexed to a radius of less than 200mm. Obviously, the more the supply line is flexed the more likely fatigue can set in, with possible rupture of the inner tube.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

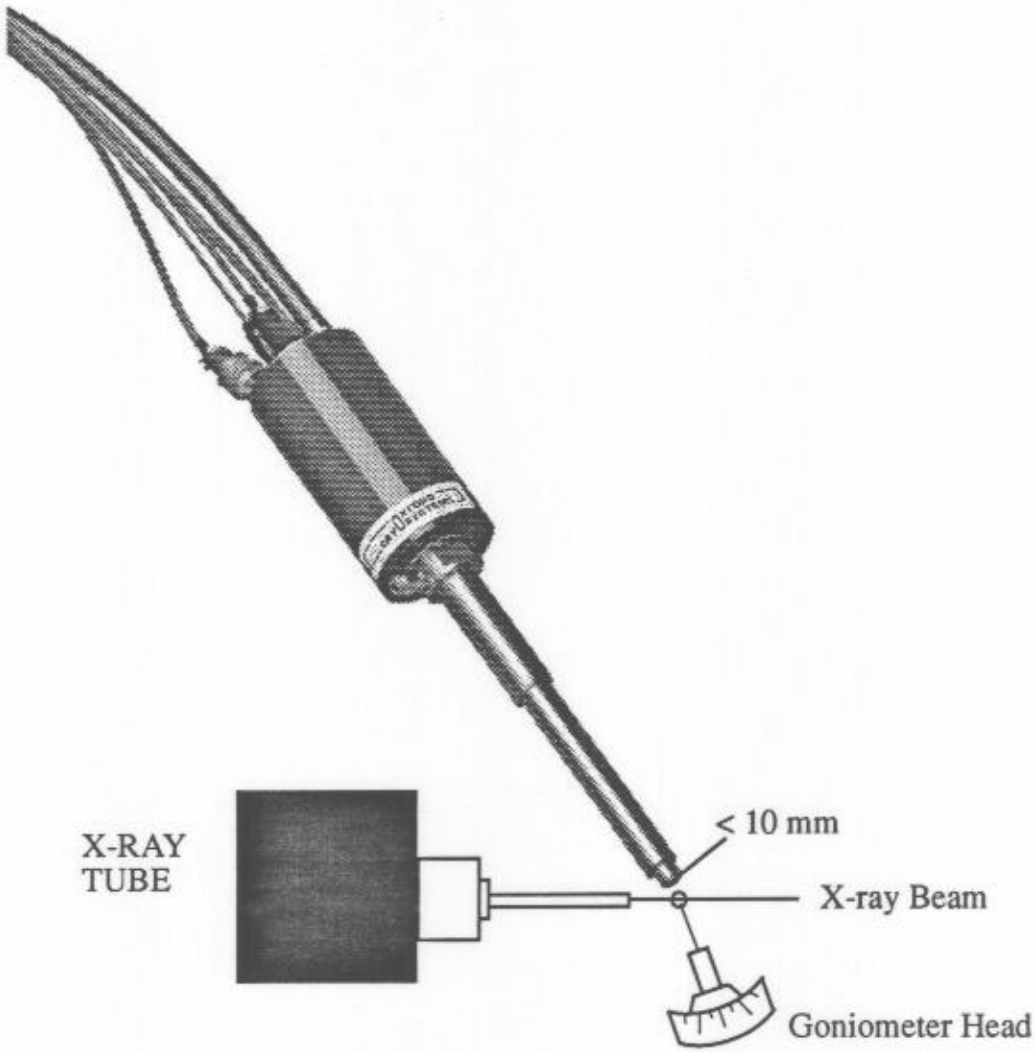
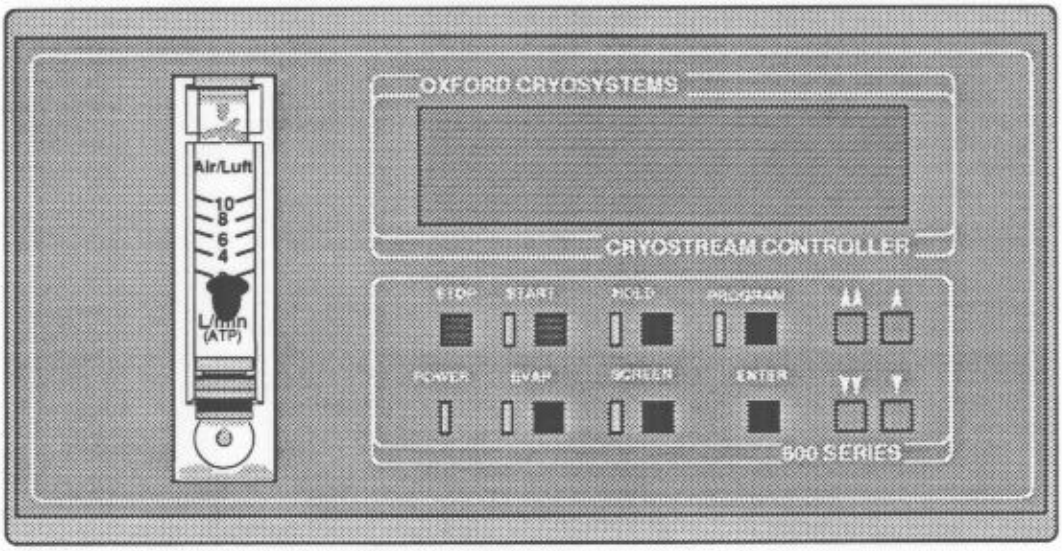
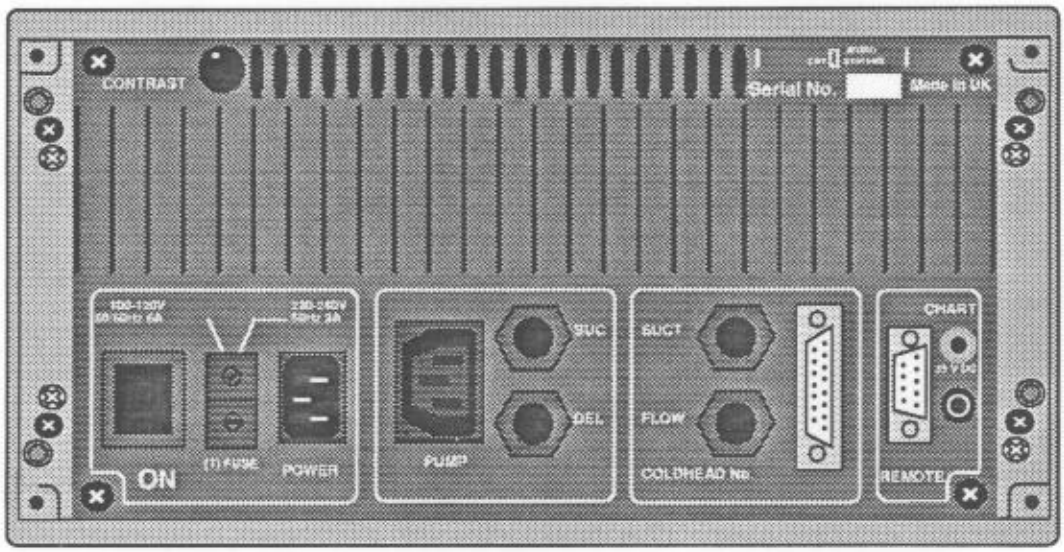


Figure 4 **CRYOSTREAM** mounting

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com



CPC611 Controller- Front View



CPC611 Controller- Back View

Figure 5

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

2.4 Connecting Up

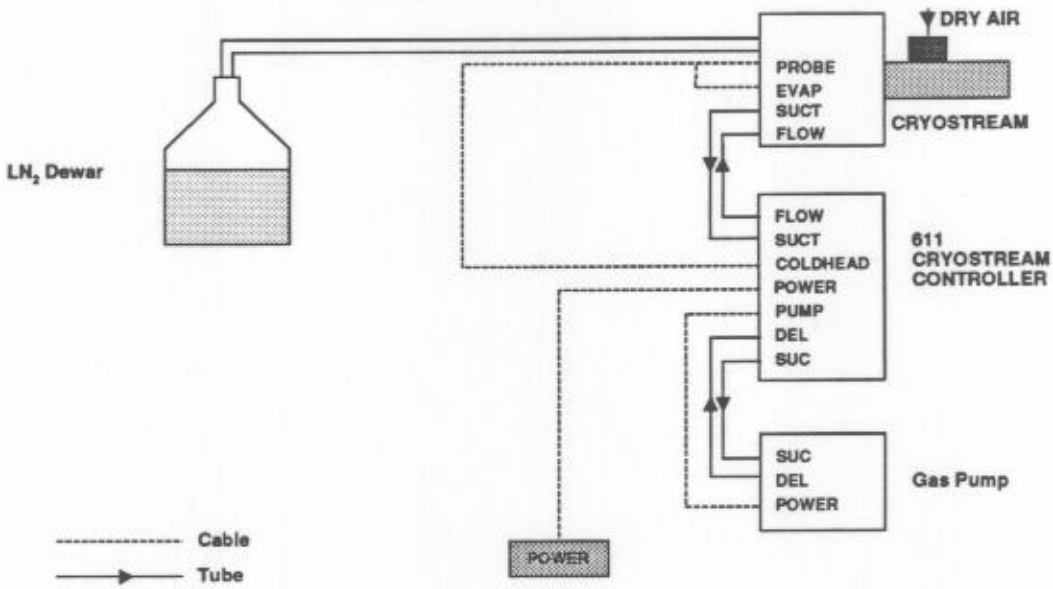
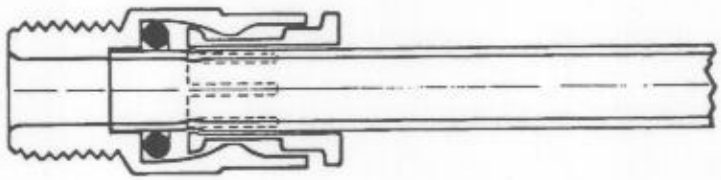


Figure 6 **CRYOSTREAM** Interconnections

Figure 6 illustrates schematically all the connections that have to be made. Connections are generally labelled equivalently at each end. Full lines denote 6mm OD nylon tubing, with arrows showing the directions of gas flow. The tube connectors are of the quick-release type. Cut the nylon tubing squarely with a sharp blade and press the end firmly into a connector until it can go no further (to release, pull the tubing whilst pressing inward the collet-ring on the connector).



Ensure that the nylon tube is inserted fully into the quick-release fitting so that the joint does not leak

Attach a nylon tube to the side connector on the delivery nozzle. This is used to pass dry air, nitrogen, etc. around the inner cold gas stream to act as a shield and to prevent icing on the crystal. Dashed lines refer to cable connections. Each cable is supplied with plugs / sockets that make them unique to avoid incorrect connections. Insert the rigid leg at the end of the flexible supply line into an open Dewar containing liquid nitrogen. You may

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

wish to press some cotton wool into the neck of the Dewar and around the supply line in order to exclude frost. However, *the Dewar must not be sealed!*

3. EVAPORATOR

In addition to the Gas Heater and Gas Temperature Sensor a second heater/sensor combination is fitted to the Evaporator (See 'E' in Fig.1, Section 1). The purpose of the Evaporator Heater is to convert most of the liquid nitrogen flowing into the **CRYOSTREAM** coldhead to vapour at its boiling point. This is necessary because the latent heat of evaporation of the liquid represents excess refrigeration of which only a fraction is needed to make up for heat leaks and heat exchanger inefficiency.

For most purposes the Evaporator is automatic and the **EVAP** key can be ignored. However, it may be necessary to adjust **EvapT** manually when operating at a Gas Temperature close to 80K (see Appendix X for details).

The temperature of the Evaporator is displayed on the Status Screen in the **EvapT** box. **EvapT** ***does not indicate the minimum Gas Temperature obtainable.***

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

OPERATION

4) CPC611 TEMPERATURE CONTROLLER

The **CPC611** temperature controller is designed to provide a completely flexible means of controlling temperature with up to about 10^{11} temperature segments. This is achieved by allowing the user to enter up to 99 phases, each one itself providing a complex series of segments. *Figure 7* below shows a typical screen with **RUN DATA** included.

265.5	Rate K/hr	Temp K	Step K	Plat mins	Loops
RAMP ↰	240	278.4			
PLAT ↰		278.4		10.0	
>STEP	120	260.0	0.1	60.0	
LOOP ↱	220	278.4			3/10
LOOP ↱					1/4

Figure 7 Typical **CPC611** screen display

Here the controller has been programmed to do the the following:

a) Starting from room temperature ramp down to 278.4K at a rate of 240 K/hour.

b) Now maintain the temperature at a plateau of 278.4 K for 10 minutes.

c) Ramp down to 260 K at a rate of 120 K/hour, but after each 0.1 K change, hold the temperature constant for 60 minutes before doing the next ramp. The > sign to the left means that this is the active phase i.e. the one that the programmer is running at the time this screen was drawn. Note that the temperature reading at the top left-hand corner 265.5 K is the actual temperature at this time.

d) Once 260 K has been reached, go back to the plateau instruction. To do this ramp to 278.4 K at a rate of 220 K/hour. This is done a total of 10 times in all. At the present time the programme is on its 3rd loop.

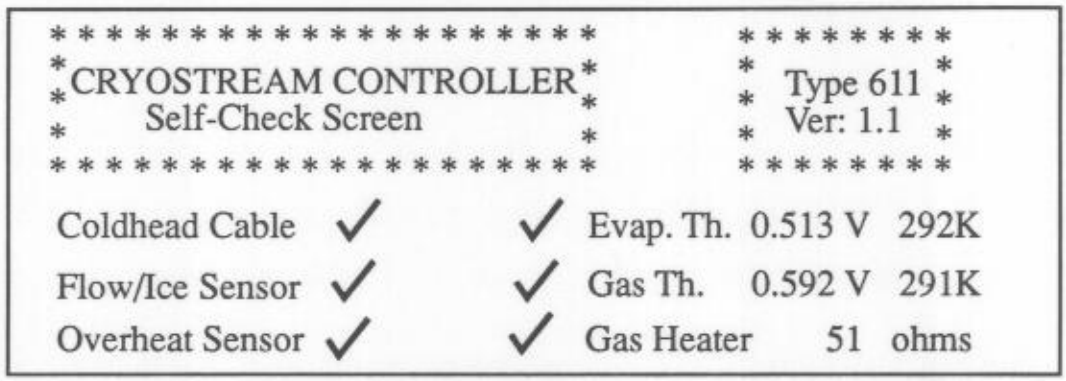
e) Finally go back to the first ramp instruction. The whole programme is rerun 4 times, and at present is on its first cycle.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

It can be seen from this example that with a few lines of programming it is possible to specify a very complex set of instructions, thus making this control system almost infinitely extensive.

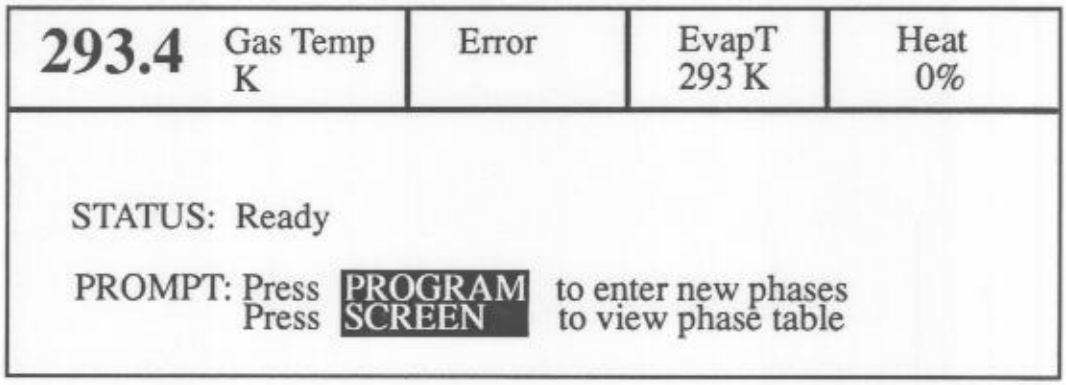
4.1 INSTRUCTIONS FOR USE

1) After connecting up and arranging a supply of liquid nitrogen, switch on the **Dry Air** supply if needed. Switch **ON** at mains and at the back of the controller. After a brief initialisation, you will observe a self-check screen similar to this:



You may need to adjust the **CONTRAST** control on the rear panel to obtain the best display on the screen. If everything is working properly, you should see each item ticked and with values similar to those indicated here (the figures will vary slightly with different Cryostreams).

2) This screen will then disappear to be replaced by a STATUS screen similar to this:



Note that room temperature should be indicating at the top left-hand corner. The **SCREEN** key allows aither the STATUS screen or the PHASE TABLE screen (which displays the program data) to be selected.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

When the screen backlight times out, pressing the **SCREEN** key switches the backlight on again.

3) Press the **PROGRAM** button in order to enter the various phases. The first phase must be a **RAMP**. After pressing the **ENTER** key you must supply the rate (range is 1 to 360K per hour) and then the final temperature (in the range 77.4 to 375K). To accept the default **RAMP** rate of 120K per hour, simply press the **ENTER** key.

4) Now with the **ARROW** keys you can select from a set of different options:

RAMP	Change temperature at a controlled rate. When ramping down in temperature, if the selected rate is too fast for the **CRYOSTREAM** to follow, the controller will automatically enter the **RAMP / WAIT** mode (this will be indicated on the screen). The effect of this is to stop the ramp in order for the gas temperature to catch up to within 5K of the gas temperature.
PLAT	Maintain temperature fixed for a certain time .
HOLD	Maintain temperature fixed indefinitely until the **START** button is pressed (a programmed **HOLD** should not be confused with the **HOLD** button)
STEP	Make a series of plateaux and ramps, each plateau being held for a certain time .
ERASE	Allow phases to be erased, one at a time, from the last one backwards, up to and including the currently active phase, which will then be replaced by a **HOLD**.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

LOOP	Allow loops to be made from the last phase to one of the preceding ones (the head of the loop can be positioned with the arrow keys). If necessary, the program requests you to ramp back to the original temperature, in order to prevent discontinuous jumps. Four nested loops are allowed. These must be completely nested; they cannot overlap. Note that the loop number **N** is the number of times that the sequence within the loop will be executed. Loops can be completely "un-nested" with the **ERASE** option.
PURGE	This allows all of the programmed phases to be removed at once. This option will only operate when the final and current phase is a programmed HOLD. If necessary, use the ERASE option to convert the final phase into a HOLD.
SLAVE	This disables most of the front panel keys and permits **CRYOSTREAM** to be controlled remotely. For more details read the section concerning the REMOTE option.
END	System shutdown. You are asked to ramp back to a final temperature and then everything is shut down. This is the controlled way to finish an experiment and should be used whenever possible.
EXIT	Exit from loading phases .

Note that not all these options will be available at any one time. The controller will only allow those options that are possible.

5) To start controlling, press the **START** button. The gas pump will start up and at first the gas flowmeter will indicate approximately 4 litres / minute and

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

rise to 5 litres / minute within 5 minutes. A typical screen during the control looks like this:

265.5 Gas Temp K	Error 0.1	EvapT 85.0 K	Heat 65%

STATUS: RAMP to 265.4 @ 120K/hr (12.5)

PROMPT: Press START to start next phase
Press SCREEN to view phase table

During the first 30 minutes of operation the gas temperature may fluctuate by ±0.3K to ±0.4K for brief intervals as the heat exchanger and evaporator cool down; avoid **RAMP**ing rapidly to below 120K during this period. The active phase is indicated by the "**>**" sign at the left of the PHASE TABLE screen(press **SCREEN** to swap STATUS/PHASE TABLE screens).

To achieve a gas temperature near 80K you may need to use **MANUAL EVAP** mode - see Appendix X for details.

It is possible to **ERASE** back any of the phases up to and including the active phase (see above). If the active phase is erased the control safely changes to a programmed **HOLD**.

It is also possible to add new phases while the temperature is being controlled. To do this press the **PROGRAM** button and add more phases. The screen automatically scrolls to the correct position to allow phases to be added as required.

6) The control program will shut down **CRYOSTREAM** if

(a) The error is greater than ±25 K.
(b) Gas flow drops below 2 l/minute* .
(c) The controller overheats.
(d) A temperature sensor (Gas temperature or Evaporator temperature) fault occurs.
(e) A coldhead cable fault is detected.

In each case the STATUS screen variables are fixed and a suitable error message is displayed.

---

* If an ice blockage starts to form in the heat exchanger an early warning BLOCK message will appear on the STATUS screen

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

7) The **STOP** button is for emergency use and will immediately shut down **CRYOSTREAM**. This can also, if required, be used if the gas temperature is close to room temperature, although a programmed **END** maybe more convenient.

8) The **HOLD** button may be pressed at any time, and will temporarily suspend the active phase, maintaining the temperature at its current value until the **HOLD** button is again pressed, whereupon the active phase will continue from where it stopped. The **START** button is used to restart after a programmed **HOLD** phase.

9) After **CRYOSTREAM** has been switched off it is advisable to leave the liquid nitrogen leg in the LN_2 Dewar (e.g. until the next day) so that the adsorption pump can maintaina good vacuum while the heat exchanger warms up gradually. This will prevent condensation or ice forming on the blue vacuum case which may cause nuisance to surrounding equipment.

10) Cold start. If **CRYOSTREAM** is started whilst still cold (after say a power interruption) the gas temperature can drop briefly, but rapidly, before proper control is established. To prevent the system shutting down due to a large error, diagnostic 6(a) above is disabled for 1 minute after start-up if **EvapT** is < 250K.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

5) TROUBLESHOOTING

Symptom	Cause	Remedy
Condensation or ice on outside of Cryostream body.	Loss of vacuum.	Repump the vacuum (see Appendix VI).
Unable to reach or maintain low gas temperature.	Loss of vacuum.	Repump the vacuum (see Appendix VI).
Low rate of gas flow.	Ice blockage due to poor quality liquid nitrogen. (A BLOCK message on the STATUS screen will often give early warning of an ice blockage).	Change liquid nitrogen supply or insert a line drier into the SUCTION line (a line drier can be obtained from Oxford Cryosystems). Clean out Dewar and refill. Cover the neck properly so that ice cannot fall into the liquid nitrogen. You can dry the **CRYOSTREAM** out by pumping dry air (possibly from the Dry Air Unit) up through the leg for several hours.
Low rate of gas flow.	Ice blockage due to leak of atmospheric air.	Locate leak. Check all pipe connections are made. Check pump diaphragm for a split.
Ice forming on one side at the end of the cold gas exit nozzle.	Dry-air shroud is not concentric with nozzle.	Realign (see Appendix II).

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Ice forming all round the end of the cold gas exit nozzle	Incorrect dry air flow	Adjust flow rate to 6-8 l/minute.
Ice forming all round the end of the cold gas exit nozzle	Dry air is contaminated with water	Service Dry Air supply
Ice forming on crystal sample or fibre	Turbulence or incorrect dry-air flow rate	Adjust dry-air flow rate to match cold stream flow.
Gas temperature is unstable	Gas temperature sensor has become pushed to one side of exit nozzle.	Inspect inside exit nozzle to see if sensor is in centre. Contact supplier if realignment is required
Gas temperature is unstable	Evaporator is set too low in MANUAL mode	Raise **EvapT** setting or return to AUTO mode
Gas temperature is unstable	Electrical interference	If it is found that **CRYOSTREAM** operates correctly away from the diffractometer, look for earth current loops or some sort of RF interference.
Evap or Gas sensor error on power up.	Bad initialization.	Switch off, wait 5 seconds, switch on again.

Note Further information can be found in the Appendices.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

APPENDICES

I. THE "ICING" PROBLEM

It is well-known that in open-stream cooling systems icing up of the crystal sample at low temperatures can be a severe problem. With the **CRYOSTREAM**, the formation of ice can be largely overcome, depending on the type of equipment that it is being used on. The dry air shroud forms a laminar flow sheath around the cold nitrogen gas stream in order to prevent damp atmospheric air causing a build-up of ice on the exit nozzle or on the crystal. You may wish to experiment with attaching Mylar film to the tip of the nozzle. One way is to cut enough Mylar to make a small cylinder and wrap it around the inner nozzle. An outer sheath of Mylar can be attached to the outside shield tube with an elastic band or with tape.

However, we have found by experience that often it is not necessary to use any Mylar sheaths, provided that care is taken with the outer stream once it is understood how the ice forms in the first place. We recommend that the outer stream should be as dry as possible (the AD31 Dry Air Unit built by **Oxford Cryosystems** is ideal for this as it is a small portable compressor that supplies air with a dew point lower than -60°C). It is important to adjust the flow rate of the outer stream to be as low as possible (typically 5-10 litres / minute) in order to match the flow rate of the inner nitrogen stream and thus not cause turbulence. Turbulence, once it occurs, brings in moist air from outside to the cold regions thus allowing ice to be deposited on the crystal or on its glass fibre mount. It is a good idea to make the glass fibre as long as possible to keep the bulky goniometer head out of the stream.

Even if there is no turbulence, ice may form at the boundary layer between the inner and outer streams because the sudden change in temperature at this point stimulates ice formation. In this case, we have found a useful trick is to mount the glass fibre inside a thin metal tube with about 1-2mm of glass sticking out at one end and to which the crystal is attached in the usual way. The metal sheath then passes through the boundary layer and because of its high conductivity this brings in sufficient heat to raise the temperature of the boundary layer just at the place where it is needed. Nevertheless, in our experience, provided that the glass fibre is thin enough, we have rarely needed to employ this technique.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

II. REALIGNING THE DRY AIR SHROUD

The dry air shroud tube is a thin-wall stainless steel tube 20 mm diameter mounted around the cold gas exit nozzle.

The shroud tube must be truly circular and concentric with the exit nozzle in order to guarantee a symmetrical air flow.

The shroud tube is locked into its 26 mm diameter mounting bush using a low strength retainer compound (e.g 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 exit nozzle - this prevents the shroud tube from kinking. Refit the shroud tube using a little retainer compound and check that the tube is concentric and allows the retainer to set.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

III. HOW CAN YOU KNOW THE TRUE TEMPERATURE AT THE CRYSTAL?

The absolute measurement of temperature is generally very difficult, especially in the heterogeneous environment of an open cold stream. The flow rate in the **CRYOSTREAM** has been designed to produce laminar flow for a few millimetres from the nozzle tip. Therefore, in order to have the crystal at a temperature close to that indicated on the controller, we advise wherever possible to ensure that the crystal sample is within 10 mm of the nozzle.

We do not recommend measuring the temperature with a thermocouple placed in the stream. In the heterogeneous environment of a narrow cold gas stream, there are several factors that lead to spuroius voltages on the thermocouple, creating errors in apparent temperature of possibly tens of degrees! For instance, conduction of heat down the wires creates a heat leak. At the point of entry of the thermocouple wires into the stream a cold junction is formed whose temperature is much lower than the room temperature assumed by the controller, thus making the measured temperature *appear* to be much higher than indicated. Also, the sharp temperature change at the interface between the cold stream and the surrounding warm air can induce stresses into the thermocouple wires and then generate spurious E.M.F.'s. We believe that the only satisfactory way to find the error in absolute temperature at the crystal position is to calibrate with a sample that undergoes a known phase transition or change of state. For instance, we have found that the low -temperature phase transition in the langbeinite $(NH_4)_2Cd_2(SO_4)_3$ was observed from intensity measurements to be in the range 88-89K (established elsewhere to be at 88K). Similarly, lattice parameter measurements of sodium ammonium tartrate tetrahydrate gave a transition temperature in agreement to within 0.5K of the published value of 109K. A most useful compilation of transitions in hundreds of crystals has been published by P. Tomaszewski (*Phase Transitions*, **38**, 127).

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

IV. REMOTE INTERFACE

This allows 611 controller to operate in **SLAVE** mode on instructions received via the **REMOTE** port.

CRYOSTREAM must be started up manually in the normal way before selecting **SLAVE** mode. It is important to ensure that **CRYOSTREAM** is functioning correctly, the **DRY AIR** flow is set correctly and the liquid nitrogen is replenished regularly.

STATUS responses can be obtained from the **REMOTE** port at any time the 611 controller is switched on (**CRYOSTREAM** off, running manually or in **SLAVE** mode). Thus temperature and error data can be logged automatically.

Properties of the SLAVE phase

The **SLAVE** phase is entered into the phase table in the same manner as any other phase. However, because its final temperature is indeterminate, no phases may be added beyond a **SLAVE** (although an existing **SLAVE** may obviously be deleted in the normal way and other phases added).

When in **SLAVE** mode, the controller executes a **HOLD** at the current set temperature until it receives a legal command request which it then executes. If another legal command request is received before the first command is complete, the first command is simply overwritten and the new one instigated. In this way running **RAMP**s may be replaced by **HOLD**s or by **RAMP**s with different parameters.

A **SLAVE** phase can be terminated either locally by pressing **PROG** and choosing **LOCAL**, or remotely by issuing a command request to leave **SLAVE** mode. In either case the **SLAVE** phase is deleted from the phase table and replaced with a **HOLD** at the current set temperature. The controller is then "back to normal", and more phases may be added and executed in the normal way - another **SLAVE** phase can be selected if required.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Communication

The controller understands, and responds with, packets of the form:

identifier<:data:data:...data:>$

Note that all data items are surrounded by colons and the whole packet terminated by a dollar sign. Two packets are recognised at all times, namely **S$** for status request, and **V$** for version request.

The response packets have the form:

Status response: S:S/L:TS:E:TF:RR:$

Here **S** is the status response identifier, the next field (i.e. the first data field) contains a single letter - either **S** for **SLAVE** mode or **L** for **LOCAL** mode, **TS** is the current set temperature, **E** the current error, **TF** and **RR** the final temperature and ramp rate of the current phase.

Version response: V:Version string:B:Baud rate:$

Here V is the version response identifier and the other fields are self-explanatory.

When the controller is in SLAVE mode a third type of packet is recognised - the command request.

Command request: C:R/S/H/L:<TF:RR:>$

Here **C** is the command request identifier. The next field contains a single letter: **R** for a simple **RAMP**, **S** for a **RAMP** followed by a **SHUTDOWN**, **H** for **HOLD** at the current temperature, **L** for leave **SLAVE** mode. The other fields are meaningful only for the first two commands: **TF** is the final temperature and **RR** the ramp rate of the requested **RAMP.**

When a command request is received an acknowledgment will be issued with the identifier **A**:

Acknowledgment: A:0/1:$

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

The data field contains either **0** to indicate that the packet was not a legal one and has been ignored (also issued in response to illegal packets received in **LOCAL** mode), or **1** to confirm receipt of a valid request which has been instigated.

When the requested command has been completed, a completion packet with identifier **D** (for "done") is issued:

Completion: D:0/1:$

The data field contains either **0** to indicate failure (**SLAVE** mode was terminated locally before the command was completed) or **1** for successful completion.

REMOTE PORT

The REMOTE connection is situated on the rear panel of the 611 **CRYOSTREAM** Controller and consists of a 9-way "D" plug with pin connections as follows:

PIN No.	FUNCTION
2	Receive Data (Rc)
3	Transmit Data (Tx)
5	Signal Earth (Gnd)

N.B. The REMOTE port is a partial implementation of RS232 and some difficulty may be experienced in making the communication link work. In this case it may help to link **Data Terminal Ready**, **Data Set Ready** and **Carrier Detect** and also to link the **Ready To Send** and **Clear To Send** at the host RS232 end of the cable.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

RS232 Settings:

Baud Rate	9600
Data Bits	8
Stop Bits	1
Parity	NONE

NOTE: The **REMOTE** port may be tested with the 611 controller switched on but **CRYOSTREAM** not running - send a version request (**V$**) or a status request (**S$**) to evoke the correct output from the 611 controller.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

V. CHART OUTPUT

The **CHART** output of the 611 **CRYOSTREAM** Controller gives a continuous analogue signal proportional to the **Error** display. **Error** is the difference between the required gas temperature and measured **Gas Temp.**

The signal level is ±5 Volts D.C which represents an **Error** of ±5K.

Connection is to two 4mm sockets on the rear panel. A Digital-to-Analogue Convertor is used for this output so that the signal varies in steps of 19.5mV or approximately 0.02K. The **CHART** output has an impedance of 1KΩ and is short-circuit protected.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

VI. PUMPING DOWN

From time to time you may need to repump the vacuum insulation space of the **CRYOSTREAM** coldhead (the blue can). This will be apparent when the outside of the coldhead becomes excessively cold or wet all over during operation.

Do not be mislead by similar symptoms which are not due to a vacuum problem. For instance, an incorrect setting of the **EVAPORATOR** or **GAS FLOW** controls could cause the end flange of the coldhead (where the electrical and gas connections emerge) to become icy. Similarly, if the liquid nitrogen supply runs low or when the nitrogen leg is removed from the storage vessel the operation of the built-in cryopump is defeated and the coldhead may become cold or damp externally.

It has been established that the **CRYOSTREAM** vacuum can be repumped using a good rotary vacuum pump (with air ballast valve), a Pirani gauge and the **CRH25** reactivation heater. It should not be necessary to use a diffusion pump.

The correct procedure is as follows:

1) Determine that the unit really does require repumping as described above.

2) Ensure that the **CRYOSTREAM** is switched off and take the nitrogen leg out of the storage vessel.

3) *If the **CRYOSTREAM** has been used recently allow at least 24 hours for the unit to warm up internally.*

4) Place the end of the nitrogen leg into the hole in the hot block of the **CRH25** heater. Ensure that nothing else can come into contact with the heater to cause damage.

Do not switch the heater on at this stage

5) Connect a good rotary pump (preferably 2-stage) to the **CRYOSTREAM PUMP-OUT PORT** using the **PUMPING ADAPTOR** (*Figure 8*) supplied.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Screw the adaptor onto the pump-out port (**VV** in *Figure 8*) positioned at the top of the rigid leg of the delivery tube.

Do not withdraw the sealing plug at this stage.

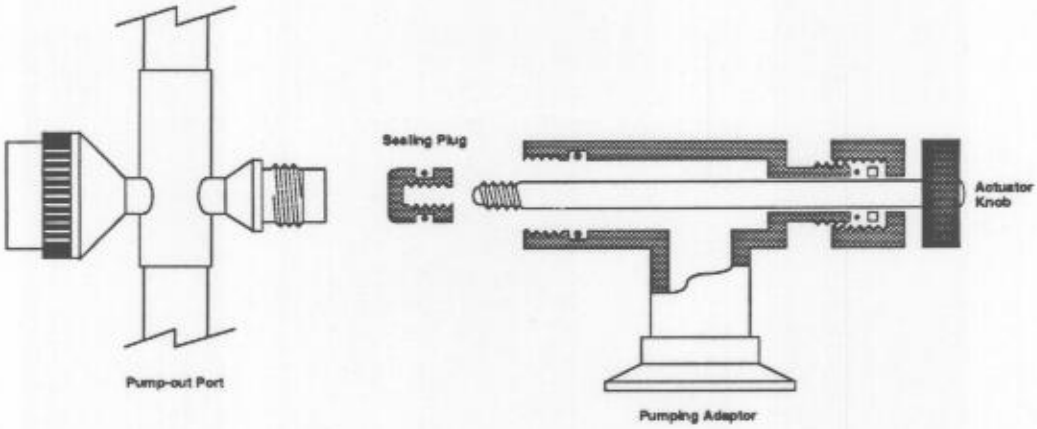


Figure 8 Attaching the Pumping Adaptor

6) Start the rotary pump and ensure that a pressure of 0.1mbar can be obtained up to the pumping adaptor. It may be necessary to run the pump with its air ballast valve open for about 30 minutes.

7) When the rotary pump pressure is 0.1mbar, use the **PUMPING ADAPTOR ACTUATOR KNOB** to locate and withdraw the **SEALING PLUG** very slowly to avoid a rush of gas. An extra valve in the pumping line would help in this operation.

8) Wait until the pressure falls towards 0.1mbar - this may take 60 minutes. If necessary, use the rotary pump air ballast again. If you cannot obtain a sufficiently low pressure at this stage you may have a leak (or bad pump!) which should be investigated. Contact your **CRYOSTREAM** supplier if all else fails.

9) When a pressure near to 0.1mbar is obtained, the **CRH25** reactivation heater can be switched on. The heater raises the temperature of the cryopump situated in the bottom of the nitrogen leg to 150-200ºC in order to drive off water and other condensable vapours. This is likely to cause the vacuum space pressure to rise and it may well be necessary to use the rotary pump air ballast yet again to purge the pump of these condensables.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

10) The vacuum should be pumped and baked for approximately 6 hours. The final pressure should be 0.01mbar (or better) with a 2-stage rotary pump.

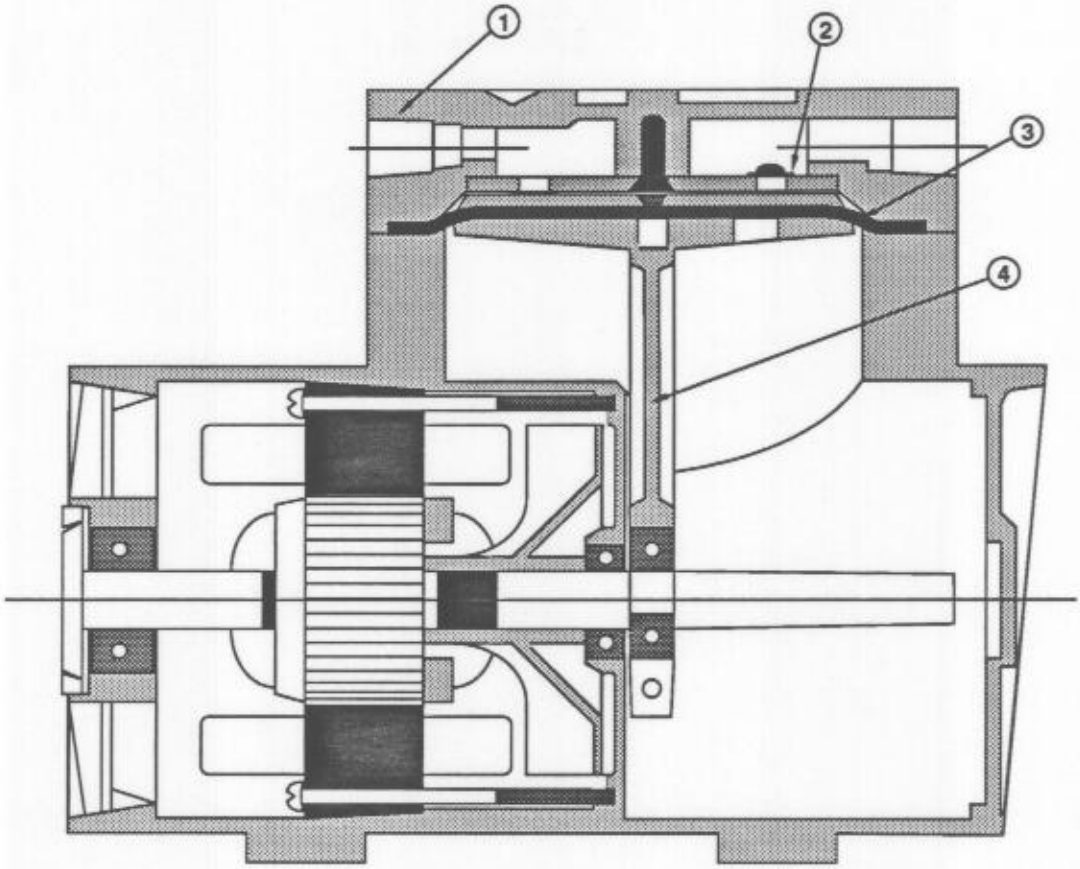
11) To finish pumping and baking, the **SEALING PLUG** should be pushed back in using the **ACTUATOR KNOB** of the **PUMPING ADAPTOR**.

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

12) Switch the **CRH25** heater off immediately and allow to cool. Unscrew the **ACTUATOR KNOB** from the **SEALING PLUG** and remove the **PUMPING PORT ADAPTOR**. Replace the **PUMPING PORT COVER.**

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

VII. THE DIAPHRAGM PUMP



No.	Part No.	Description
1	660446	Cylinder Head
2	621102	Valve Flapper
3	608144	Diaphragm
4	607213	Connecting Rod Assembly

Figure10 Thomas 905CD18 Diaphragm Pump

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

VIII. ICE BLOCKAGE

Very occasionally small quantities of water may be present in the nitrogen stream flowing through the **CRYOSTREAM**. If sufficient water is present an ice blockage will form and this can reduce the flowrate or cause a "**LOW FLOW**" trip. The blockage forms in the **FLOW** path of the coldhead as the nitrogen is recooled from room to liquid nitrogen temperature.

Two possible sources of this water are in the liquid nitrogen supply or a suction leak in the **SUCT** or **SUCTION** paths of the circuit. Irregular blockages can be caused by variable quality of liquid nitrogen, whilst increasingly frequent blockages may indicate a leak of atmospheric air into the nitrogen stream.

Try to ensure that your supply of liquid nitrogen is of the highest possible purity. Use some sort of cover where the nitrogen leg passes into the neck of the Dewar, but do not seal the Dewar. Take care to avoid contamination of the liquid nitrogen during refills.

If a leak is suspected, check that the nylon tubes penetrate each quick-release fitting by approximately 15mm. After thousands of hours running, the Gas Pump Diaphragm may fatigue and develop a leak - see Appendix II for details of fitting a replacement diaphragm.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

IX. CRYOSTREAM SERVICE

The **CRYOSTREAM** has been designed to be as easy to use as possible and should run without the need for constant attention, once you have become accustomed to its method of working. However, there are some important points that you should look out for.

1) If you find that the outer case of the cryostat becomes wet or frosty, this probably indicates a loss of vacuum. You may also find that the Gas Temperature is higher than the Set Temperature or that you have to decrease the Evaporator power in order to maintain temperature. The lower the temperature you wish to attain the more important the vacuum becomes. The remedy is to pump out the system as described in Appendix V.

2) A localized cold spot is probably not caused by poor vacuum. Ice near the SUCT connector on the Coldhead may mean an Evaporator problem (some condensation at this point is to be expected). Condensation or ice spots on the flexible line, however, may indicate that the inner liquid nitrogen capillary tube is touching the wall of the outer flexible tube due to excessive bending.

3) If the inner tube in the supply line should rupture, liquid nitrogen can then enter the vacuum region. If then for some reason the delivery tube is warmed up, say by pulling it out of the Dewar, the liquid nitrogen will evaporate to produce gas at high pressure inside the vacuum region. The safety valve fitted opposite the pump-out port is designed to vent excessive pressure to atmosphere.

*If you suspect a vacuum leak has developed, do not attempt to use **CRYOSTREAM**: contact your agent / supplier.*

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

X. EVAPORATOR

Auto Mode

This is the default mode. On start-up, when the **CRYOSTREAM** coldhead is at room temperature, **EvapT** will be approx 293K falling to 85K within 30 minutes.

At Gas Temperatures below 150K the value of **EvapT** is automatically reduced slightly to produce the extra refrigeration required.

In order to reach a Gas Temperature below 100K it may be necessary to allow 2+ hours running for the heat exchangers inside the coldhead to cool down fully.

Manual Mode

To operate at a very low Gas Temperature you may need to reduce the **EvapT** manually - this provides the extra refrigeration required to reach the lowest temperature. ***Always allow* CRYOSTREAM *to run for 2+ hours before attempting this adjustment.***

To select **Manual Mode** the **EvapT** must be <100K.

Select the Status Screen and press the **EVAP** key - the **EVAP** lamp will glow and the **EvapT** display will show the EvapT set point value highlighted as shown below:-

88.9 Gas Temp K	Error 0.1	EvapT 81.5 K	Heat 10%

STATUS: RAMP to 85.0K @ 120K/hr (4.7)

PROMPT: Press START to start next phase
Press SCREEN to view phase table

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Change the EvapT set point value using the ↑ or ↓ keys and then the **ENTER** key - the **EvapT** display will change back to normal but the **EVAP** lamp will flash to indicate **Manual Evap Mode**. ***Reduce the EvapT by 0.1-0.2K steps and allow 5-10 mins for the change to have an effect. If the EvapT is reduced too much, liquid nitrogen droplets will form in the exit nozzle gas stream and cause temperature instability of ±0.5K or greater.***

If the **EVAP** key is pressed during **Manual Evap Mode** the **EvapT** display will first change to **AUTO**, to revert to **Auto Evap Mode** press the **ENTER** key or to adjust **EvapT** to a new value press the **EVAP** key again.

Artisan Scientific - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisan-scientific.com

Artisan Technology Group is an independent supplier of quality pre-owned equipment

Gold-standard solutions

Extend the life of your critical industrial, commercial, and military systems with our superior service and support.

We buy equipment

Planning to upgrade your current equipment? Have surplus equipment taking up shelf space? We'll give it a new home.

Learn more!

Visit us at **artisantg.com** for more info on price quotes, drivers, technical specifications, manuals, and documentation.

Artisan Scientific Corporation dba Artisan Technology Group is not an affiliate, representative, or authorized distributor for any manufacturer listed herein.

We're here to make your life easier. How can we help you today?

(217) 352-9330 | sales@artisantg.com | artisantg.com

ARTISAN® TECHNOLOGY GROUP