

OptistatDN-V

Variable Temperature Liquid Nitrogen Cryostat

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Calibration and test results appear at the end of this manual.

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1 Important information

1.1 Warnings

Before you attempt to install or operate this equipment for the first time, please make sure that you are aware of the precautions that you must take to ensure your own safety.

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 Temperature and voltage limits

If you have bought a cryostat and temperature controller together from Oxford Instruments the temperature controller will have been set up in the factory:

- To prevent you from accidentally exceeding the maximum safe operating temperature of the cryostat
- To limit the maximum heater voltage to a safe level

If you are planning to use an existing temperature controller, or a power supply or controller made by another manufacturer, you should take the same precautions. The recommended values for the 'Heater Voltage Limit' and the 'Temperature Limit (T_{HOT})' are given with the test results for the cryostat.

Caution: If you do not safeguard the system it is possible to cause serious damage.

1.3 Important Note

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.

1.4 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.

1.5 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.

1.6 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 Introduction

2.1 Introduction to this manual

The OptistatDN-V is a liquid nitrogen cryostat designed principally for

- Optical spectroscopy
- Electrical measurements requiring optical access

The sample temperature is continuously variable between 77 K and 500 K.

This manual is designed to describe the system and explain how to operate it with the Oxford Instruments ITC502 or ITC503 temperature controller. We recommend that you read this manual carefully before you try to operate the system. With practice you will find the best way to use the system for your own special application.

2.2 Safety requirements

Please refer to the separate booklet *Safety Matters* which we have supplied with the system. This includes information about the properties of liquid nitrogen, and detailed recommendations about the precautions you should take. It is your responsibility to ensure your own safety, and the safety of the people working around you

3 Description of the system

3.1 The system

Figure 1 is a schematic diagram of the OptistatDN-V cryostat, showing the main features.

The liquid nitrogen is stored in a reservoir which surrounds the central sample access tube but is thermally isolated from it. Liquid is supplied to the sample space heat exchanger through a capillary tube. The flow of liquid is aided by gravity, but the flow rate is controlled by the exhaust valve at the top of the sample space. A 100 Ω platinum resistor and heater have been fitted to the sample space heat exchanger, and the temperature controller is used to supply the required amount of heat to balance the cooling power and set the required temperature.

The sample is positioned directly below the heat exchanger, and up to five windows can be fitted to give optical access to the sample space.

The reservoir and sample space are thermally isolated from the room temperature surroundings by the outer vacuum chamber (OVC). This space is pumped to a high vacuum before the cryostat is cooled down, and the vacuum is maintained by a small sorption pump fitted to the reservoir. This continuously pumps the residual gases from the OVC to maintain good thermal isolation. A heater has been fitted to the sorb, and this is used to drive the adsorbed gases out of the activated charcoal when the system is at room temperature. The sorb should be re-activated whenever the OVC is pumped.

The optical path to the sample space has an angular aperture of f 1.0, where the f number is defined as:

$$f \text{ number} = \frac{\text{distance from centre of sample space to OVC}}{\text{diameter of aperture on OVC}}$$

The windows are sealed into the OVC by rubber 'O' rings and they can be removed easily.

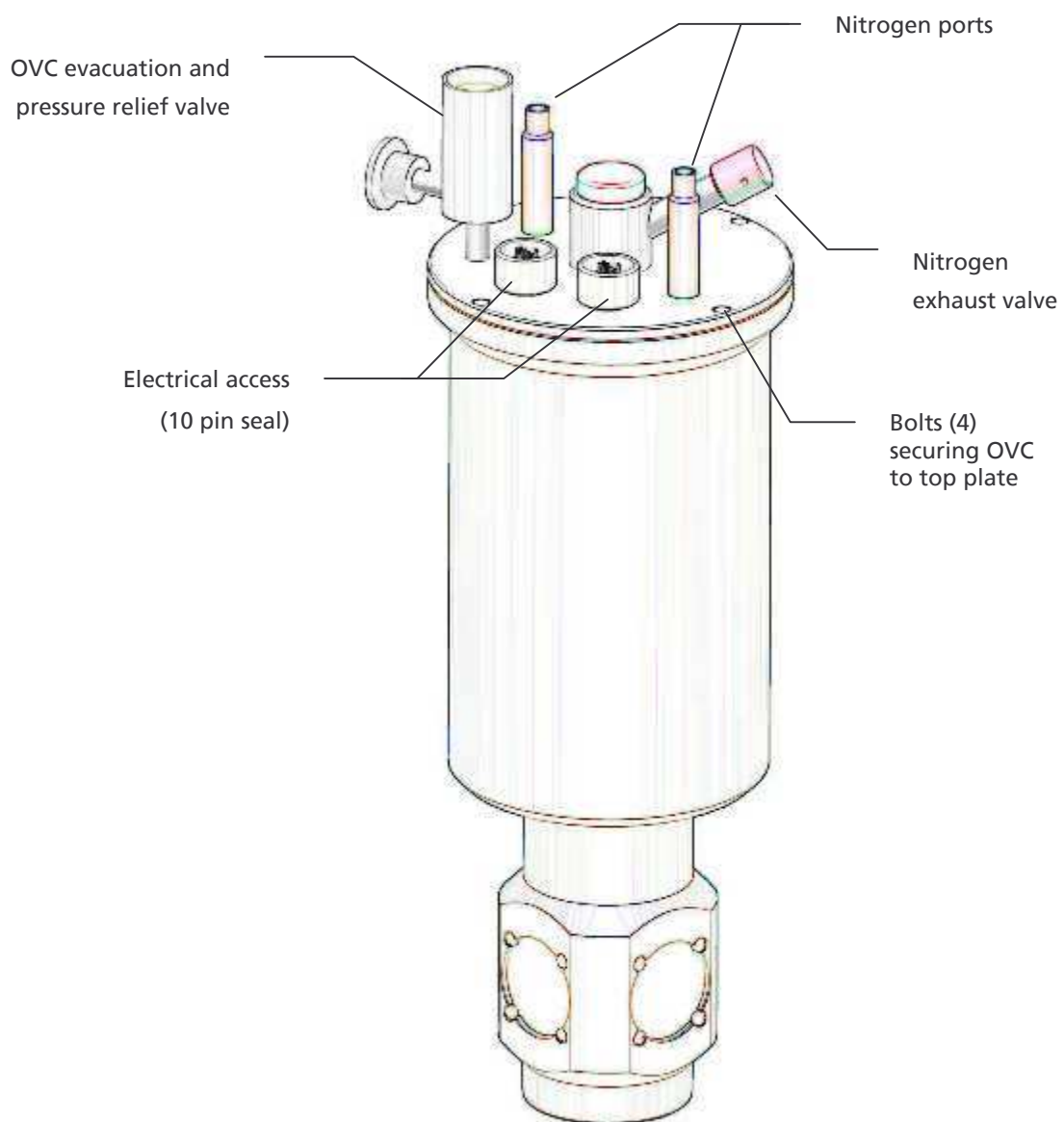


Figure 1 Schematic diagram of OptistatDN-V cryostat

4 Unpacking and preparation

4.1 Unpacking the system

Carefully remove the cryostat and all the accessories from the packing case, 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.

4.2 Preparing the system for operation

Choose a suitable position to operate the cryostat safely, and if necessary arrange for it to be supported so that it cannot accidentally fall. Use Figure 1 to help you to locate the services and controls on the top plate of the cryostat.

4.2.1 Loading the sample

Vent the cryostat by slowly opening the evacuation valve on the top plate. Remove the four bolts which secure the OVC to the top plate, and remove the OVC. Fix the sample to the sample holder, and connect the required wiring. Replace the OVC and the four retaining bolts.

4.2.2 Evacuating the OVC

The OVC has to be pumped to high vacuum (lower than 10^{-4} mbar) to make sure that it gives the required thermal isolation. When the system is new, all 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, just that the new materials are being cleaned by the vacuum. The sorb will pump these gases effectively, but it has a limited capacity and it has to be re-activated regularly. The OVC should be pumped thoroughly before each cooldown, especially when the cryostat is new.

Connect the pumping system to the cryostat vacuum valve on the top plate of the cryostat. We recommend that you use a diffusion pump or turbo-molecular pump, backed by a rotary pump, and fitted with a cold trap which helps the system to pump water vapour. If the system is badly contaminated with water vapour, the gas ballast facility on the rotary pump should be used.

4.2.3 Activating the sorb

The sorb should be activated before you cool the system. This helps to maintain the quality of the vacuum in the OVC. If the sorb is not re-activated it is likely that it will become saturated quickly, and the cryostat boil-off will soon become unacceptably high.

The normal cryostat cable is used with a special adapter to outgas the sorb. Connect the CC1 cryostat cable to the temperature controller. Connect the SORB adaptor cable to the ten pin seal on the cryostat top plate, and connect the two leads together. This connects the temperature controller to the heater on the sorb, not the heater on the sample space heat exchanger.

Evacuate the OVC as described in section 4.2.2. Press and hold the heater MAN button on the temperature controller, and use the RAISE and LOWER buttons to adjust the heater voltage to 12 V. This is shown on the main display of the temperature controller while the MAN button is held down. When the button is released, the heater output is shown graphically on the heater display. Leave the sorb to outgas with the heater voltage at 12 V for at least 4 hours, and preferably overnight.

Make sure that the heater voltage is not set above 12 V as this could damage the cryostat. After the sorb is reactivated, allow it to cool for at least 120 minutes before you close the valve on the cryostat and disconnect the pump.

5 Running the system

5.1 Cooldown

5.1.1 Preparations

Remove the SORB adaptor lead from the ten pin seal and connect the grey or black cable directly to the cryostat. This makes the necessary connections to allow the temperature controller to monitor the thermometer on the heat exchanger, and supply current to the heater on the sample space heat exchanger. Switch on the temperature controller. The main display should now show the temperature of the sample space heat exchanger. If the heater is on (as shown by the LEDs on the heater display), press and hold MAN and LOWER to switch it off.

Ensure that the OVC has recently been pumped to high vacuum and that the sorb has been activated as described in section 4.2.3. Open the gas exhaust valve (needle valve) on the cryostat top plate, by 3 or 4 turns.

Set the 'set temperature' of the temperature controller to below 77 K, by pressing and holding the SET button, and using RAISE/LOWER to adjust the set point shown on the main display.

5.1.2 Cooling the cryostat

Caution

If the vacuum is accidentally lost in the OVC while the cryostat is cold then this will cause the cryostat to freeze. This will not damage the system.

Warning

Before you start to use liquid nitrogen make sure that you are aware of the precautions that are necessary to ensure your safety.

Put a suitable funnel into one of the liquid nitrogen vent ports on the cryostat. Put a filter (such as a piece of paper towel) into the funnel. This helps to remove frozen particles from the liquid nitrogen and reduce the risk of blockages in the heat exchanger capillary tube. Fit a short length (say 10 to 20 cm) of suitable polythene tube over the other ports to direct the cold gas away from the top plate and to stop it freezing the 'O' ring. Slowly pour liquid nitrogen into the reservoir. The liquid will boil violently at first, and then suddenly the boil-off will drop. Fill the reservoir until liquid comes out of the other vent tube.

The temperature of the sample space heat exchanger (which is shown on the temperature controller) should now begin to drop. This should settle at about 77 K. When the heat exchanger reaches base temperature, close the exhaust valve slightly to reduce the liquid nitrogen consumption. If this valve is closed too much, the temperature will begin to rise again.

Since quite a large amount of the liquid nitrogen from the reservoir has been used to cool the heat exchanger to base temperature, you should now refill the reservoir, so that the cryostat has a long hold time at base temperature.

5.2 Operation at base temperature

Once the cryostat has been cooled to base temperature the exhaust valve may need to be adjusted slightly, as the liquid level in the nitrogen reservoir drops. Refill the liquid nitrogen reservoir occasionally to keep a constant supply of liquid to the heat exchanger. If the system is to be operated for a very long time, the sorb may become saturated, and this will be indicated by water condensing on the outside of the OVC. If this happens, the OVC should be pumped continuously to maintain the required high vacuum.

Caution: Do not try to re-activate the sorb while the cryostat is cold, as this would evaporate all the liquid nitrogen. You must warm the sorb to above room temperature to re-activate it properly.

5.3 Temperature control above base temperature

5.3.1 Introduction

You can control the temperature of the sample space heat exchanger (and the sample) between base temperature and 500 K. The flow of liquid nitrogen and the heater power have to be adjusted to reach the required set point. The ITC502 or ITC503 temperature controller is used to control the heater power automatically, and adjusts the power to maintain the set temperature. These temperature controllers are three term controllers. The temperature control is optimised by setting the best values for:

- Proportional band (P)
- Integral action time (I)
- Derivative action time (D)

The values given in the test results for the system are suitable to give good stability. If you want to improve the stability further you may be able to do this by adjusting the three terms slightly. The procedure for optimising the PID values and control theory are given in the temperature controller manual.

5.3.2 Controlling at a 'set temperature'

Check that the cryostat has been connected to the temperature controller as described in section 5.1.1

Close the exhaust valve (needle valve) fully, and then open it by quarter to half a turn. This sets the flow of liquid to the heat exchanger at a suitable level, but the flow can be optimised later.

Set the required 'set temperature' by pressing and holding the SET button on the temperature controller, and using the RAISE/LOWER buttons to adjust the value shown on the main display. Set the PID values to those shown for the nearest temperature in the test results. Press the AUTO button once, and the temperature controller should adjust the heater output to warm the heat exchanger to the 'set temperature'.

It is not necessary to cool the cryostat to base temperature before you set the required 'set temperature'. If the temperature controller is set to the required temperature at the beginning of the cooldown, the cryostat should cool to the set temperature and the temperature controller should then hold it at this point.

You should then optimise the flow of liquid nitrogen so that the heater output of the temperature controller is not too high. In general, the flow should be reduced until the steady heater output is less than 12 V.

5.3.3 Operation above room temperature

Pump the OVC continuously to maintain the required high vacuum. You can now control the system at a 'set temperature' as described in section 5.3.2

5.4 Changing samples

Warning Before you change sample make sure that you take all necessary precautions to avoid getting cold or hot burns.

The sample can only be changed by warming the whole cryostat to room temperature, and following the procedure given in section 4.2.1.

5.5 Warming up the system

If you do not need to warm the system quickly it may be left to warm up naturally. If you want to warm it more quickly, pour the liquid nitrogen out of the reservoir and allow a small volume of dry nitrogen gas into the OVC to break the vacuum.

6 Maintenance

If you carry out a few simple procedures your cryostat will give you years of reliable operation.

6.1 Rubber 'O' rings

Whenever you remove part of the cryostat or if you suspect that there is a leak on the system check the 'O' rings on the sample space and OVC for cuts or abrasions. If you find a damaged ring, replace it.

6.2 Evacuating the OVC and re-activating the sorb

The OVC will need to be pumped regularly and the sorb should be re-activated as described in section 4.2.3.

6.3 Window replacement

If you need to clean the windows, they can be removed as follows.

The windows are shown in Figure 2. Ensure that the cryostat OVC is at atmospheric pressure. Carefully remove the four screws that hold the window in place. If the cryostat is turned over, the window will fall out. Clean it with a suitable lens cleaner and lens tissue. Before the window is replaced, check the inner 'O' ring, clean it, and grease it lightly. After you have replaced the 'O' ring place the window on top and tighten the four nylon screws.

Note The nylon screws only need to be finger tight. Do not overtighten as this may damage the window or the screws.

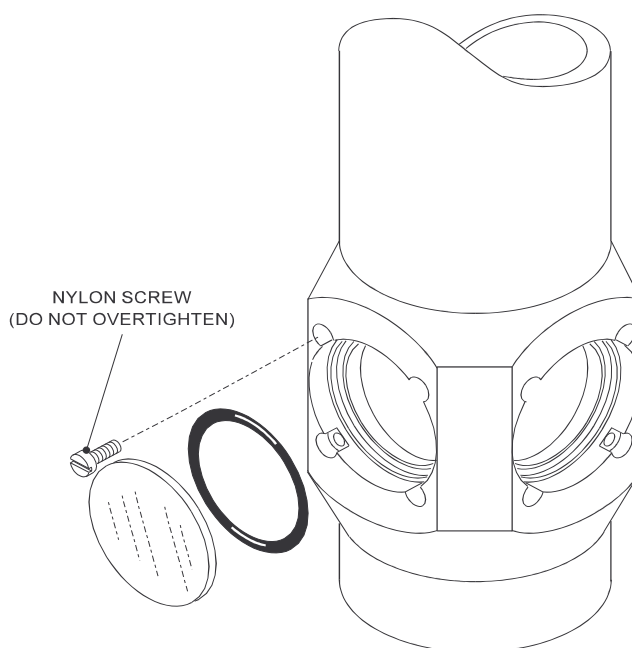


Figure 2 'O' ring sealed outer windows

7 Electrical connections on the cryostat

The standard cryostat is fitted with a ten pin seal on the top plate. This is used for the connection to the sample space heat exchanger and the sorb. The seal is held in place by the black knurled nut. Do not remove it unless you need to gain access to the wiring.

The heat exchanger is fitted with a thermometer (set up for four wire measurement) and a heater. Detailed information about the thermometer is given in section 9.

Pin	Function	
A	Heat exchanger heater (nominal 20 ohms)	
B		
C	V+	Heat exchanger platinum thermometer
D	V-	
E	I+	
F	I-	
H	Spare	
J	Spare	
K	Sorb heater	
L		

Note An additional 10 pin connector is fitted to the top plate of the cryostat and wired if requested when the system was ordered. The wires from this connector are usually terminated just above the sample holder.

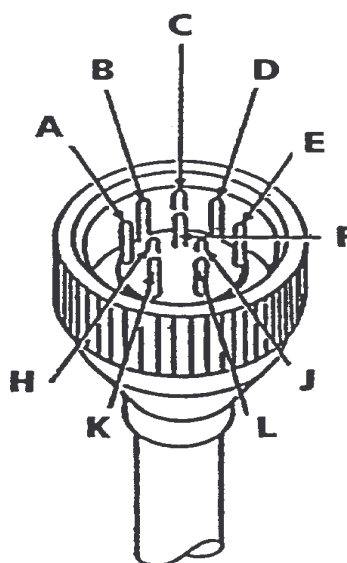


Figure 3 Ten-pin seal terminals

8 Fault finding

Symptom	Diagnosis and suggestions
<p>Cryostat OVC cannot be pumped to high vacuum</p> <p>Or</p> <p>Water condenses on the cryostat body when it is cold</p>	<p>Check that the sorb is not full and outgassing slowly, by re-activating as described in section 4.2.3.</p> <p>Or</p> <p>Check the cryostat OVC for leaks. In particular check:</p> <ul style="list-style-type: none"> • Cryostat top plate seals • Outer window seals
<p>Cryostat will not cool down</p>	<p>Check whether there is any flow of gas from the exhaust valve. If necessary, bungs may be fitted to the liquid reservoir exhaust ports temporarily so that the pressure is slightly increased while you check the exhaust. Remove them immediately after the test, and remember to wear gloves when you do this.</p> <p>If the sample space capillary is blocked, warm it up and blow dry gas through it.</p>
<p>Cryostat cannot be warmed up from base temperature</p>	<p>Check that the heater is not open circuit by checking from pin A to pin B.</p>
<p>Sensor not reading correctly</p>	<p>Check the wiring</p> <p>Check that the (black or grey) adaptor lead has been removed</p>

9 Platinum Resistance Thermometer

9.1 Description

The Platinum Resistance Thermometer is a low cost industrial grade miniature temperature sensor, suitable for cryogenic use over the range 70 to 900 K. It is the ideal choice where temperature measurement or control is required to liquid nitrogen temperatures.

The sensor conforms to BS 1904:1984 Class A band 4 with the temperature/resistance relationship shown in Table 2. Class A band 4 refers to the manufacturing resistance tolerance of the sensor.

9.2 Specifications for 100 ohm Platinum resistance thermometer

Part No.	PRZ0015
Temperature Range	70-900 K
Resistance	100 ohms at 273 K, 138.50 ohms at 373 K
Stability/year	130 mK
Reproducibility	60 mK
Nominal resistance at 0°C	100 Ω
Calibration available	No

Table 1 Specifications

9.3 Variation of resistance with temperature

Temperature		Resistance
C	K	Ohms
-200	73	18.49
-190	83	22.80
-180	93	27.08
-170	103	31.32
-160	113	35.53
-150	123	39.71
-140	133	43.87
-130	143	48.00
-120	153	52.11
-110	163	56.19
-100	173	60.25
-90	183	64.30
-80	193	68.33
-70	203	72.33
-60	213	76.33
-50	223	80.31
-40	233	84.27
-30	243	88.22
-20	253	92.16
-10	263	96.09
0	273	100.00
10	283	103.90
20	293	107.79

Temperature		Resistance
C	K	Ohms
30	303	111.67
40	313	115.54
50	323	119.40
60	333	123.24
70	343	127.07
80	353	130.89
90	363	134.70
100	373	138.50
110	383	142.29
120	393	146.06
130	403	149.82
140	413	153.58
150	423	157.31
160	433	161.04
170	443	164.76
180	453	168.46
190	463	172.16
200	473	175.84
210	483	179.51
220	493	183.17
230	503	186.82
240	513	190.45

Table 2 BS1904 resistance vs temperature characteristics