Oxford Instruments NanoScience

Optistat DN-V

Operators Manual

System Manual / Issue 2.0 / December 2020

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Contents

1 Introduction

This manual is designed to introduce you to the Optistat DN-V, manufactured by Oxford Instruments. This manual contains important information for the safe operation of your system. You must read this manual before operating the system for the first time.

In addition to this manual for the Optistat DN-V, further manuals and documentation will have been supplied with the system. These additional manuals and documents detail the other components of the system, as well as important safety information, as shown in Table 1-1. Please ensure you have reviewed the information supplied in all the manuals before you attempt to operate your system.

Documentation	Format
MercuryiTC manual	Electronic copy on USB
Practical cryogenics	Electronic copy on USB
Safety matters	Electronic copy on USB
Optistat DN-V QuickStart guide	Electronic copy on USB
Optistat DN-V test results	Electronic copy on USB
Optistat DN-V safety sheet	Electronic copy on USB
MercuryiTC safety sheet	Hard copy

Table 1-1: Documentation supplied with the Optistat DN-V.

Please keep all the manuals supplied with your system and make sure that you regularly check for updated information and incorporate any amendments. If you sell or give away the product to someone else, please give them the manuals too.

These are the Original Instructions.

1.1 Copyright

technical inaccuracies or typographical errors. Changes are periodically made to the information contained herein; these changes will be incorporated in new editions of the document.

1.2 Statement of intended use

The equipment has been designed to operate within the process parameter limits that are outlined in the user manual. The equipment is intended to be installed, used and operated only for the purpose for which the equipment was designed, and only in accordance with the instructions given in the manual and other accompanying documents. Nothing stated in the manual reduces the responsibility of users to exercise sound judgement and best practice. It is the user's responsibility to ensure the system is operated in a safe manner. Consideration must be made for all aspects of the system's life-cycle including, handling, installation, normal operation, maintenance, dismantling, decontamination and disposal. It is the user's responsibility to complete suitable risk assessments to determine the magnitude of hazards.

The installation, usage and operation of the equipment are subject to laws in the jurisdictions in which the equipment is installed and in use. Users must install, use and operate the equipment only in such ways that do not conflict with said applicable laws and regulations. If the equipment is not installed, used, maintained, refurbished, modified and upgraded as specified by the manufacturer, then the protection it provides could be impaired. Any resultant non-compliance damage, or personal injury would be the fault of the owner or user.

Use of the equipment for purposes other than those intended and expressly stated by Oxford Instruments, as well as incorrect use or operation of the equipment, may relieve Oxford Instruments or its agent of the responsibility for any resultant non-compliance damage or injury. The system must only be used with all external covers fitted.

1.3 Restrictions on use

The equipment is not suitable for use in explosive, flammable or hazardous environments. The equipment does not provide protection against the ingress of water. The equipment must be positioned so that it will not be exposed to water.

1.4 Maintenance and adjustment

Only qualified and authorised persons should service or repair this equipment. Under no circumstances should the user attempt to repair this equipment while the electrical power supply is connected.

1.5 Warranty

The Oxford Instruments customer support warranty is available to all our customers during the first 12 months of ownership from date of delivery. This warranty provides repair to faults that are a result of manufacturing defects at Oxford Instruments NanoScience.

1.6 Acknowledgements

All trade names and trademarks that appear in this manual are hereby acknowledged.

1.7 Technical support

If you have any questions, please direct all queries through your nearest support facility (see below) with the following details. Please contact Oxford Instruments before attempting to service, repair or return components.

System type: Optistat DN-V
Serial number: The Sales Order (SO) number and/or other identifiers of your system.
Contact information: How we can contact you, email/telephone details.
Details of your query: The nature of your problem, part numbers of spares required, etc.

Europe, Middle East, Africa and India (EMEAI)

OINS, Tubney Woods, Abingdon, Oxon, OX13 5QX, UK
Tel: +44(0)1865 393200 (sales)
Tel: +44(0)1865 393311 (support)
Fax: +44(0)1865 393333 (sales and support)
Email: nanoscience@oxinst.com (sales)
Email: ServiceNSUK@oxinst.com (service and support)
Web: www.oxford-instruments.com

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Email: ServiceNSAsia@oxinst.com (service and support)
Web: www.oxford-instruments.cn

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Fax: +81 3 6732 8939 (sales and support)
Email: nanoscience.jp@oxinst.com (sales, service and support)
Web: www.oxford-instruments.jp

2 Health and safety

Before you attempt to install or operate your system, please make sure that you are aware of all safety precautions listed in this manual together with the warnings and cautions set out in other documents supplied with the system.

All cryogenic systems are potentially hazardous, and you must take precautions to ensure your own safety. The general safety precautions required when working with cryogenic systems are given in Oxford Instruments' *Safety Matters* document. We recommend that all users should read this document, become thoroughly familiar with the safety information provided and be aware of the potential hazards.

It is the responsibility of customers to ensure that the system is installed and operated in a safe manner. It is the responsibility of customers to conduct suitable risk assessments to determine the nature and magnitude of hazards.

2.1 Disclaimer

Oxford Instruments assumes no liability for use of any document supplied with the system if any unauthorised changes to the content or format have been made.

Oxford Instruments' policy is one of continued improvement. The company reserves the right to alter without notice the specification, design or conditions of supply of any of its products or services. Although every effort has been made to ensure that the information in this document and all accompanying documents is accurate and up to date, errors may occur. Oxford Instruments shall have no liability arising from the use of or reliance by any party on the contents of this these documents (including this document) and, to the fullest extent permitted by law, excludes all liability for loss or damages howsoever caused.

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

2.2 Disposal and recycling instructions

You must contact Oxford Instruments (giving full product details) before any disposal begins. It is also important to check with the appropriate local organisations to obtain advice on local rules and regulations about disposal and recycling.

2.2.1 WEEE

Oxford Instruments Nanotechnology Tools Ltd is a scheme member for end of product life disposal.

The scheme is operated by:

B2B Compliance, Emerald House, Cabin Lane, Oswestry, Shropshire, SY11 2DZ
Tel: 01691 676124
Fax: 0808 280 0468
E-Mail: info@b2bcompliance.org.uk
Web: www.b2bcompliance.org.uk

2.2.2 RoHS compliance

All the materials and components used in the manufacture of the Optistat DN-V are in compliance without exemption with the EU Directive 2011/65/EU for Restrictions of Hazardous Substances (RoHS). This is based on information provided by Oxford Instruments suppliers and is accurate to the best of our knowledge.

2.3 Maintenance

Observe the necessary maintenance schedule for the system. Consult Oxford Instruments if you are unsure about the required procedures. Only qualified and authorised persons must service or repair this equipment.

2.4 General hazards

The following general hazards must be considered when planning the site for installation and operating the equipment. Please take notice of the following relevant warnings.

2.4.1 Warning notices

Warning notices draw attention to hazards to health. Failures to obey a warning notice may result in exposure to the hazard and may cause serious injury or death. A typical warning notice is shown below.

WARNING
A warning triangle highlights danger which may cause injury or, in extreme circumstances, death.

2.4.2 Caution notices

Caution notices draw attention to events or procedures that could cause damage to the equipment, may severely affect the quality of your measurements, or may result in damage to your sample or measurement apparatus. Failure to obey a caution notice may result in damage to the equipment. A typical caution notice is shown below.

CAUTION
Caution notices highlight actions that you must take to prevent damage to the equipment. The action is explained in the text.

2.5 Specific hazards

Safety information that applies specifically to the Optistat DN-V is provided in this manual. Where additional components are supplied as part of a system, please read and follow all safety information in the respective manuals and take additional precautions as necessary.

2.5.1 Hazardous voltages

HAZARDOUS VOLTAGE
Contact with hazardous voltage can cause death, severe injury or burns. Ensure that a local electrical earth (ground) connection is available at the installation site.

PROTECTIVE EARTH

The cryostat and any other parts of the system fitted with earthing points must always be connected to protective earth during operation.

Parts of the system carry high voltages that can cause death or serious injury. Ensure that a local electrical earth (ground) connection is available.

The electrical supply to the system must include an isolation box to ensure that mains electrical power to the system can be isolated. The isolation box must allow the supply to be locked OFF but must NOT allow the supply to be locked ON.

2.5.2 Low temperatures

COLD OBJECTS

Contact with cold objects and cryogens can cause serious injury to the skin. Skin may adhere to cold objects. Ensure that any cryogenic or coolant delivery systems are designed to prevent contact with the cold components.

Consider the hazards of low temperatures when planning the installation of the system. Proper safety equipment, including hand and eye protection, must be made available to all personnel expected to handle cryogenic liquids.

2.5.3 Pressure relief

CLOSED VESSELS

Closed vessels in the system are protected by pressure relief valves that exhaust directly to atmosphere unless otherwise stated.

Do not tamper with any of the pressure relief devices fitted to the system or attempt to modify or remove them. Also ensure that the outlets of the relief devices are not obstructed. The correct operation of these relief valves is critical to the safety of the system. All closed vessels in the system are protected by pressure relief valves, as described in Table 2-1.

Location	Description	Setting
Outer Vacuum Chamber	Relief valve to atmosphere	0.25 bar differential

Table 2-1: Pressure relief valve information.

The system's pumping valve for the outer vacuum chamber has in-built pressure relief plates, as shown in Figure 2-1. This allows the system's vacuum chamber to vent to atmosphere if it becomes over-pressurised. A restoring spring provides the force required to re-seal the cap (red) automatically when the pressure drops.

Figure 2-1: The pressure relief valve on the Optistat DN-V.

Do not modify or tamper with these safety features in any way. Additionally, ensure that nothing can restrict the movement of any of the pressure relief valves. The relief valves should not vent during normal operation of the system.

2.5.4 Weight and lifting

HEAVY OBJECT

Incorrectly lifting heavy objects can cause severe injury. Use appropriate lifting equipment, operated by fully trained personnel, when handling heavy system components.

Appropriate lifting equipment and Personal Protective Equipment (PPE) must be provided for the duration of the system installation and should always be used whilst operating or moving the system.

2.5.5 Asphyxiation

ASPHYXIATION

Nitrogen can displace the oxygen from air and cause death by asphyxiation. Ensure that adequate ventilation is provided.

Areas where these chemicals are stored or used must be well ventilated to avoid the danger of asphyxiation. Oxygen level detection equipment should be installed in suitable locations to warn personnel if the oxygen concentration falls below a threshold value. Take precautions to prevent spillage of liquid cryogens.

2.5.6 Fire

FLAMMABLE GAS

Atmospheric oxygen can condense on cryogenically cooled objects. Oxygen can cause flammable substances to ignite in the presence of heat or arcing, risking severe injury.

Rooms where cryogenic liquids are being handled must be designated as no smoking areas. While liquid helium and nitrogen do not support combustion, their low temperature can cause oxygen from the air to condense on surfaces and may increase the oxygen concentration in these areas. Oxygen enrichment may cause spontaneous combustion.

2.5.7 Trip hazards

TRIP HAZARDS
Poorly routed cables and pumping lines can be trip hazards and have the potential to cause accidents. Such accidents can result in both damage to the system and injury to personnel.

Where cables and lines are required, their routings should be considered when planning the installation of the system. The cables and pumping lines of the system should be routed away from walkways and away from areas of common use to prevent the hazards.

2.5.8 Slip hazards

SLIP HAZARDS
During normal operation, ice may form on parts of the system. Upon warm up, this ice may melt and pool by the system. Water on the floor has the potential to cause accidents. Such accidents can result in both damage to the system and injury to personnel.

Drip trays should be placed appropriately around the system to catch any water run-off. Additionally, warning signs should be placed around the system.

2.5.9 Temperature and voltage limits

The Optistat DN-V system is supplied with a MercuryiTC temperature controller. Safety features for the temperature controller are described in the MercuryiTC manual supplied with the system. You should ensure that you understand and comply with all safety warnings and cautions.

The MercuryiTC will have been set up in the factory to prevent you from accidentally exceeding the maximum safe operating temperature of the cryostat, and to limit the heater voltage to a safe level. If you are planning to use an existing temperature controller, or a controller made by another manufacturer, you should take the same precautions. The recommended values for the temperature controller limits are shown in Table 2-2.

Control Limit	Control Value
Heater voltage	40 V
System temperature	510 K

Table 2-2: MercuryiTC system control limit values

TEMPERATURE & VOLTAGE LIMITS
If you do not safeguard the system with control limits, it is possible to cause serious damage to the system.

2.6 Safety equipment

The following items are recommended for safe operation of any system:

- Personal protective equipment, including thermally insulated gloves, face protection, body protection and protective footwear. Cryogens can act like water, soaking into clothing and causing severe burns.

- Hazard warning signs, barriers or controlled entry systems to ensure that personnel approaching the system are aware of the potential hazards. This precaution is especially important if your system includes a superconducting magnet.
- Oxygen monitors should be fitted in the laboratory to warn personnel if the concentration of oxygen in the air falls below safe levels.

2.7 Risk assessments

It is the responsibility of customers to perform their own risk assessments before installing, operating or maintaining the system. Risk assessments must obey regulations stipulated by the local regulatory authority.

3 System description

The Optistat DN-V is a liquid nitrogen cryostat and can be held at temperatures between 77 K and 500 K using a MercuryiTC temperature controller. A sample can be mounted on a sample holder which is thermally linked to the system's heat exchanger providing cooling during operation. Up to five windows can be fitted to the Optistat DN-V providing optical access to the sample holder with an optical *f* number of 1.0.

The cryostat's liquid nitrogen is stored in a reservoir, which surrounds the central heat exchanger support tube, but is thermally isolated from it. Liquid nitrogen is supplied to the heat exchanger through a capillary tube connected to the reservoir. The flow of liquid is aided by gravity, but the flow rate is controlled by the exhaust valve at the top of the cryostat. The heat exchanger is fitted with a sensor and heater, and the MercuryiTC temperature controller is used to balance the cooling power and heater voltage to set the required temperature.

The reservoir and sample holder 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 sorb continuously pumps the residual gases from the OVC to maintain good thermal isolation. The sorb is fitted with a heater which is used to drive the adsorbed gases from the activated charcoal when the system is at room temperature.

A range of window materials are available for the Optistat DN-V, covering most of the electromagnetic spectrum.

3.1 The Optistat DN-V cryostat

A schematic of the Optistat DN-V is shown in Figure 3-1. The main features of the cryostat are:

- The outer vacuum chamber (OVC) with four radial windows and one axial window.
- A liquid nitrogen reservoir.
- The nitrogen exhaust flow needle valve.
- System heat exchanger with heater and sensor.
- A pressure relief device on the OVC.
- A diagnostic 10-pin connector for temperature control.
- An experimental 10-pin connector for experimental measurements.
- An experimental harwin pin ring above the sample holder.

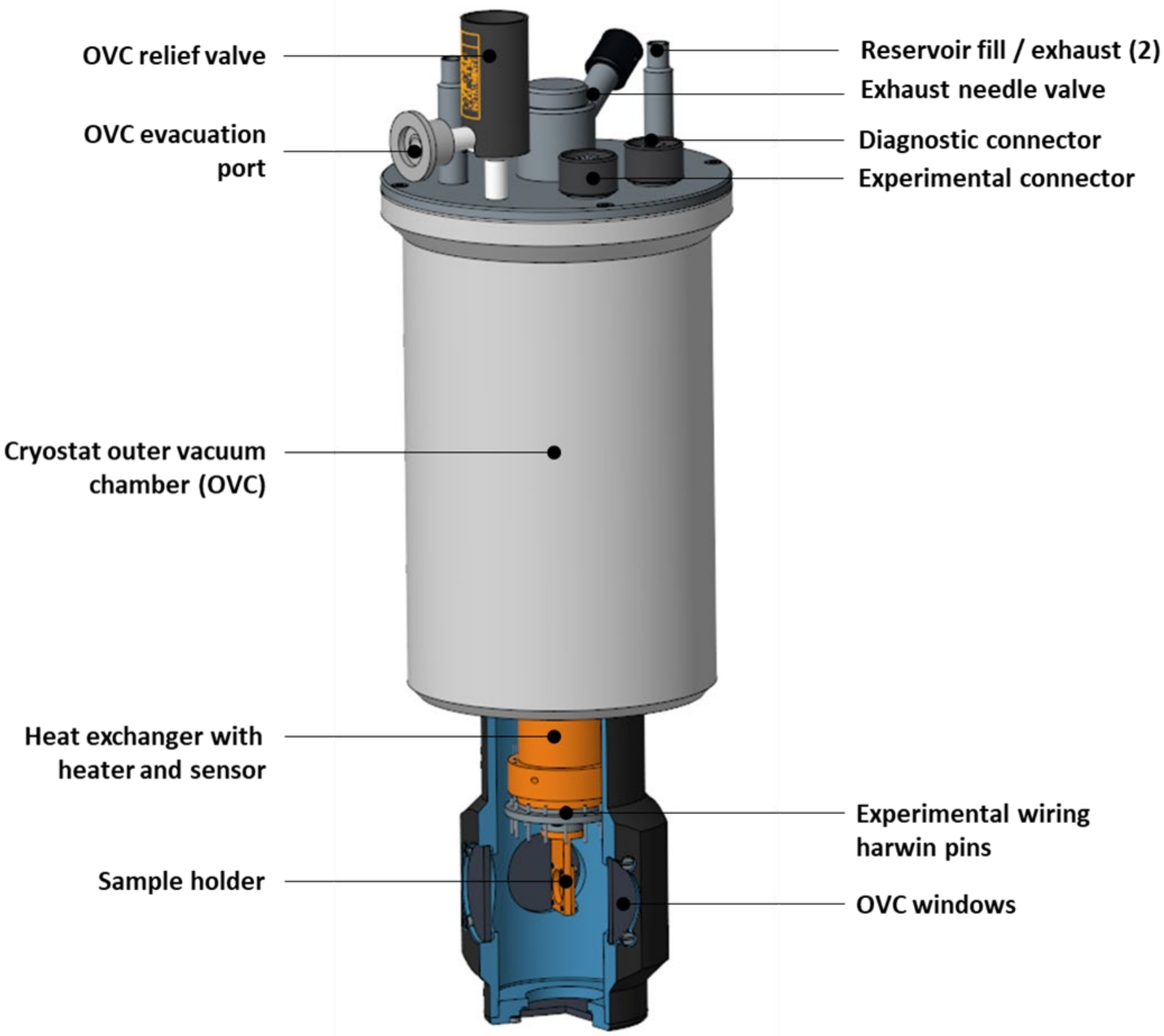

Figure 3-1 Optistat DN-V cryostat schematic.

3.2 Sample holders

There are two sample holders supplied with the Optistat DN-V. The first is a plain reflectance sample holder, as shown in Figure 3-2 (Left), several small samples can be mounted on to this sample holder at once. The second is a transmission sample holder, as shown in Figure 3-2 (Right), which has a 15 mm diameter aperture and includes a sample retaining clamp.

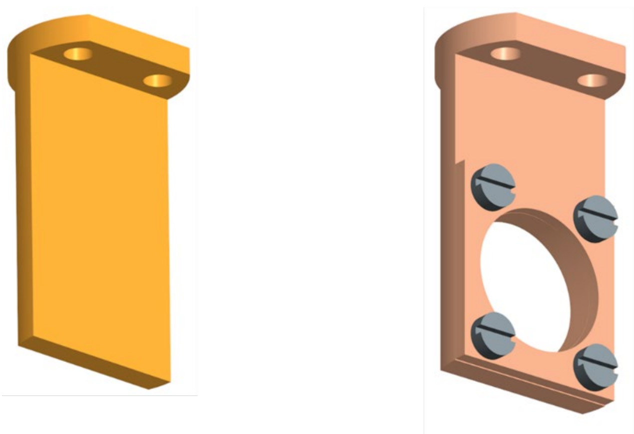

Figure 3-2: Optistat DN-V reflectance (Left) and transmission (Right) and sample holders.

3.3 The MercuryiTC temperature controller

A MercuryiTC is used as the temperature controller for the system. The MercuryiTC monitors and controls the thermometry of the system and adjusts the system's heater voltage to hold the sample holder at a defined temperature.

The MercuryiTC is configured with measurement cards in specific locations. This configuration is detailed in Table 3-1.

Slot	Card Type	Function	Connection
Main Board	Sensor & Heater	Temperature Control	Diagnostic 10-Pin
1-8	Not used	n/a	n/a

Table 3-1: MercuryiTC configuration for the Optistat DN-V.

3.4 System wiring

The Optistat DN-V cryostat is fitted with two 10-pin connectors on the OVC top plate. One is used for system control and the other is for experimental wiring. The diagnostic connector is used for the connection to the sample space heater and sensor as well as the system's sorb. The pin configuration for the 10-pin diagnostic connector is shown in Figure 3-3. The seal is held in place by a black nut, this nut should not be removed, unless access to the wiring is required.

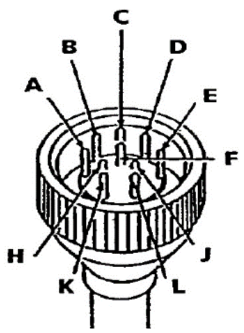

Figure 3-3: Pin configuration for the 10-pin connector.

The wiring configuration for the diagnostic connector is set out in Table 3-2, as per the pin configuration shown in Figure 3-3.

Pin	Function	Polarity	Type
A	Heat Exchanger Heater	H+	Firerod heater (20 Ω nominal)
B		H-	
C	Heat Exchanger Sensor	V+	Platinum resistance thermometer (100 Ω nominal)
D		V-	
E		I+	
F		I-	
H	Not Used	N/A	N/A
J			
L	Sorb Heater	S+	Sorb heater (150 Ω nominal)
K		S-	

Table 3-2: Pin configuration for the control and diagnostic 10-pin connector.

The experimental connector is wired to the Harwin pin ring above the sample holder. The pin configuration for the 16-pin Harwin pin ring is shown in Figure 3-4. On the Optistat DN-V, pin one of the Harwin pin ring is denoted by a black dot.

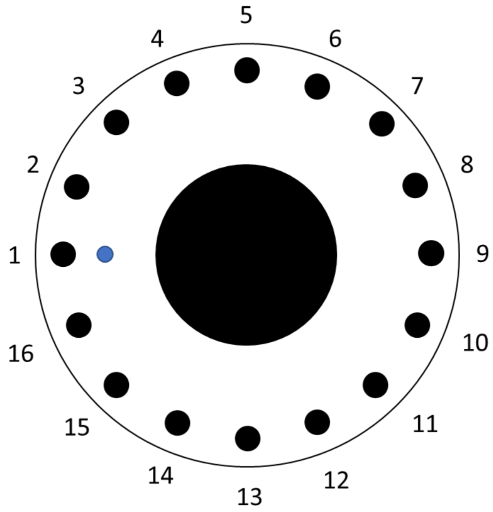

Figure 3-4: Pin configuration for the 16-pin Harwin pin ring.

The wiring configuration for the experimental connector is set out in Table 3-3, as per the pin configurations shown in Figure 3-3 and Figure 3-4.

Connector Pin	Harwin Ring Pin	Wire
A	1	34 SWG Cu
B	2	34 SWG Cu
C	3	40 SWG Cu
D	4	40 SWG Cu
E	5	40 SWG Cu
F	6	40 SWG Cu
H	7	40 SWG Cu
J	8	40 SWG Cu
L	9	40 SWG Cu
K	10	40 SWG Cu
-	11 - 16	No connection

Table 3-3: Pin configuration for the experimental 10-pin connector.

3.5 Sensor calibrations

The calibrations for the calibrated sensor on the system, a platinum resistance thermometer, will have been loaded onto the MercuryiTC in the factory when tested. The platinum resistance thermometer is an industrial grade miniature temperature sensor, suitable for cryogenic use over the range of 70 K to 900 K. It is the ideal choice where temperature measurement or control is required down to liquid nitrogen temperatures.

The sensor conforms to BS 1904:1984 Class A band 4, with the temperature/resistance relationship shown in Table 3-4. Class A band 4 refers to the manufacturing resistance tolerance of the sensor.

Specification	Value
Temperature Range	70 - 900 K
Stability/year	130 mK
Reproducibility	60 mK
Nominal Resistance	100 Ω at 273 K 138.5 Ω at 373 K

Table 3-4: Platinum resistance thermometer temperature / resistance relationship.

File "RP51.dat" is provided with the system information on your USB stick. This file contains the generic calibration data for the PT100 sensor.

3.6 System components and layout

A schematic showing the connections between the components of the system is shown in Figure 3-5

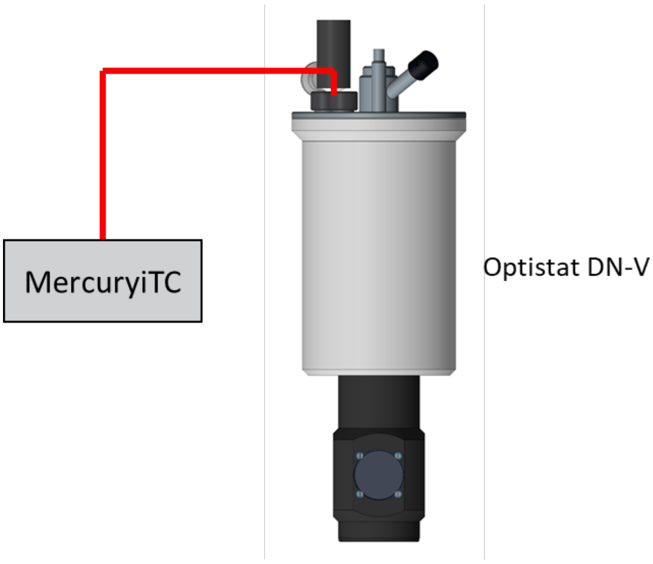

Figure 3-5: System components and layout.

3.7 Weights and dimensions

The dimensions and weights for the main system components are given in Table 3-5.

Component	Length / mm	Width / mm	Height / mm	Weight / kg
Optistat DN-V Cryostat	175	175	450	5
MercuryiTC	310	485	90	8

Table 3-5: Weights and dimensions of system components.

4 System installation

The setup of an Optistat DN-V is a straightforward procedure requiring no specialist training.

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 all 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.

To run this system, the following components are required:

- The Optistat DN-V cryostat.
- MercuryiTC temperature controller.
- Sensor & heater cable - CQB0090.
- Sorb heater cable with adaptor - CWA0123.
- A supply of liquid nitrogen (customer provided).
- High vacuum pumping system and lines (customer provided).

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.

> PRESSURE RELIEF VALVES
> The closed vessel of the system is protected by a pressure relief device. Ensure that the pressure relief device is not obstructed in any way during system setup.

4.3 Loading the sample

To load a sample, the cryostat OVC must first be removed. Follow the procedure detailed in Section 5.9 to first remove the OVC and provide access to the sample stage. The sample can now be loaded, and any desired wiring connected. If a change in sample holder is required, follow the procedure detailed in Section 5.10. Once the sample has been loaded, continue following the procedure in Section 5.9 to replace the cryostat OVC.

4.4 Evacuating the OVC

Before running the system, the OVC must be pumped to high vacuum to ensure it provides the required thermal insulation. 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. The system's sorb will pump these gases effectively; however, it has a limited capacity. Therefore, the OVC should be pumped thoroughly and the sorb should be reconditioned, as described in Section 4.6, before each cooldown, especially when the cryostat is new.

To evacuate the cryostat's vacuum space, first connect the pumping system to the DN16NW OVC evacuation port. Before you open the valve to the cryostat vacuum space, evacuate the pumping line to 10^{-4} mbar. Then open the valve by turning the relief valve cap anti-clockwise a few turns. You can then begin pumping the cryostat vacuum space to a high vacuum (lower than 10^{-4} mbar). If possible, leave it to pump-down overnight. Once pumping is complete, turn the relief valve cap clockwise to close the valve. Once the valve has been firmly closed, vent the pumping line and disconnect the pumping system from the cryostat. After the evacuation of the OVC, Oxford Instruments recommends fitting a DN16NW blanking flange to the evacuation port to prevent any possible leaks.

For the pumping system, Oxford Instruments recommends that you use a turbo-molecular pump, backed by a rotary pump, and fitted with a cold trap which helps the system to remove any water vapour. If the system is badly contaminated with water vapour, the gas ballast facility on the rotary pump should be used.

4.5 Connecting to the MercuryiTC temperature controller

The MercuryiTC has been configured by Oxford Instruments to suit the ordered system. When you first switch on the MercuryiTC, you will see the instrument home screen, similar to the screen shown in Figure 4-1.

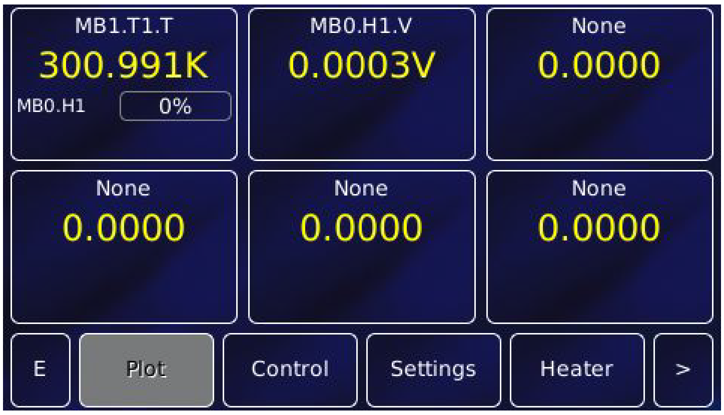

Figure 4-1: MercuryiTC home screen.

The cables from the MercuryiTC should be connected as per Table 4-1 with the MercuryiTC switched off. After the cable connections have been made, turn on the MercuryiTC. The temperature should read approximately 295 K (room temperature).

Cable	From (Mercury)	To	Function
CQB0090	MB1 (Sensor & heater)	OVC body (Diagnostic 10-pin)	Heat exchanger, sensor & heater

Table 4-1: System's wired connections.

4.6 Reconditioning the sorb

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

To recondition the sorb, the cryostat cable (CQB0090) is used with a special adapter (CWA0123). Plug the adapter CWA0123 between the controller cable and the cryostat. During the activation, 12 V is applied across the 150 Ω sorb heater using the MercuryiTC. The cryostat OVC must be pumped continuously during the sorb reconditioning process, refer to Section 4.4 for the OVC evacuation procedure.

On the MercuryiTC home screen, tap ‘Heater’. The screen shown in Figure 4-2 will be displayed.

- Tap ‘Resistance Set’ and change the resistance value to 150 Ω.
- Tap ‘Voltage’ and change this to 12 V.
- Tap ‘Home’ to return to the home screen.

Figure 4-2: MercuryiTC heater window.

On the Mercury iTC home screen, tap ‘Control’.

- Tap ‘Sweep’, followed by ‘Load’. Select the file ‘DN-Desorb’.
- Tap ‘Close’.
- Tap ‘Heater Auto’.
- Tap ‘Fixed’.

The sorb is now heated to 308 K using the sequence shown in Table 4-2.

Final Temperature / K	Time to Temperature / min	Time at Temperature / min
308	1	600
50	1	999

Table 4-2: Optistat DN-V sorb regeneration sequence.

Initially, when the heater is showing 100%, go to the MercuryiTC home screen and confirm that the voltage is 12 V. A higher voltage may damage the sorb.

Continue pumping until the pressure in the OVC falls below 10^{-4} mbar. As a default setting, after 600 minutes at 308 K, the set temperature is reduced to a value far below room temperature, at which point the heater voltage will drop to zero. This is a safety feature. After the sorb is reconditioned, allow the system to cool for at least two hours before you close the valve on the cryostat and disconnect the pump.

Remove the CWA0123 lead from the 10-pin connector and connect the controller cable (CBQ0090) 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.

During normal operation, up to 40 V is applied across the 20 Ω heat exchanger heater. Following sorb reconditioning, ensure that the voltage limit is adjusted as follows:

- On the Mercury iTC home screen, tap 'Heater'.
- Tap 'Resistance Set' and change the resistance to the value given in the test results. This will be about 20 Ω.
- Tap 'Voltage' and change this to 40 V.
- Tap 'Home' to return to the home screen.

5 System operation

This section describes the operation of the Optistat DN-V in conjunction with an Oxford Instruments MercuryiTC temperature controller. The cryostat can be operated manually if a temperature controller is not available, although it may be difficult to obtain good temperature control in this configuration.

5.1 Preparations

Ensure that the system has been properly prepared for operation, as described in Section 4.

SYSTEM PUMP DOWN
The system's OVC must be pumped thoroughly to a high vacuum before each cooldown.

WORKING WITH CRYOGENS
Before you start to use any cryogens, make sure that you are aware of the precautions that are necessary to ensure your safety. Refer to *Safety Matters* for more information.

5.2 Cooling the system

USING CRYOGENS
Before you start to use liquid nitrogen, make sure that you are aware of the precautions necessary to ensure your safety. Refer to *Safety Matters* for more information.

Open the exhaust valve fully and put a suitable funnel into one of the liquid nitrogen fill/exhaust ports on the cryostat. Fit a short length (around 1 m) of suitable polythene tube over the other fill/exhaust port to direct the cold gas away from the top plate and to stop it freezing the O-ring.

RESERVOIR VENTING PIPE
Make sure that the venting pipe is pointing in a safe direction, as liquid nitrogen will spray out the vent pipe if the nitrogen bath is over-filled.

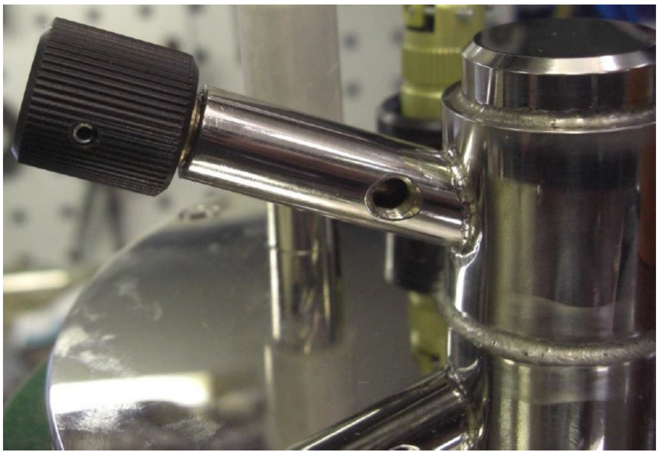

Figure 5-1: Optistat DN-V exhaust needle valve.

Slowly pour liquid nitrogen into the reservoir. The liquid will boil violently at first, before suddenly dropping off. Fill the reservoir until liquid comes out of the vent tube.

!	LIQUID NITROGEN CONTAMINATION Ensure that the nitrogen supply is free from contaminants such as frozen particles. This will reduce the risk of blockages in the heat exchanger capillary tube.

Once the cryostat is full of liquid nitrogen, the temperature of the heat exchanger, as shown on the MercuryiTC, will 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 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.

5.3 Operation at base temperature

To keep the Optistat DN-V cryostat at a stable temperature with no applied heat, open the needle valve fully.

Once the cryostat has been cooled to base temperature, the exhaust needle valve may need to be adjusted slightly as the liquid level in the nitrogen reservoir drops. Refill the liquid nitrogen reservoir occasionally to maintain a constant supply to the heat exchanger.

If the system is to be operated for a very long time, the sorb may become saturated, indicated by condensation on the outside of the OVC. If this is the case, pump the OVC continuously to maintain a high vacuum.

!	RECONDITIONING THE SORB Do not recondition the sorb whilst the cryostat is cold, as this will evaporate all the liquid nitrogen in the reservoir. The cryostat should be warmed as per Section 5.8 before reconditioning the sorb.

5.4 Temperature control above base temperature

You can control the temperature of the heat exchanger (and sample) between base temperature and 500 K. The nitrogen flow and heater power must be adjusted in order to reach the desired set point.

The Mercury iTC can control the heater power automatically, adjusting the applied power to maintain the set temperature. The Mercury iTC is a three-term controller - the temperature control is optimised by setting the best values for:

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

On the MercuryiTC home screen, as previously shown in Figure 4-1, the 'Control' button at the bottom of the screen can be selected to give the screen shown in Figure 5-2. From here, the system temperature can be set.

Figure 5-2: MercuryiTC 'Control' screen.

Tapping 'PID table' in the bottom left will display a screen like the one shown in Figure 5-3. This screen shows the current PID table loaded into the MercuryiTC. In this case, 'Optistat DN Mercury.pid' should already be loaded into the MercuryiTC.

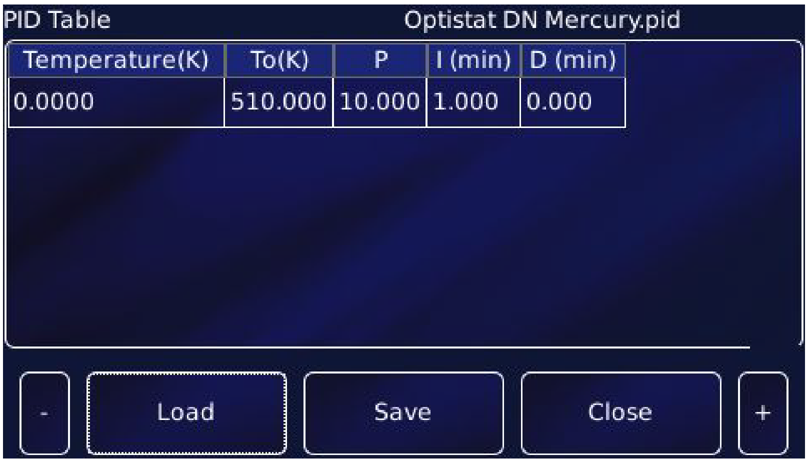

Figure 5-3: MercuryiTC Optistat DN PID table screen.

If an alternative PID table is desired, tapping 'Load' on the 'PID table' screen allows alternative PID tables to be viewed, as shown in Figure 5-4. These may be from the factory or created by the user. Tap a filename to select it and then tap 'Load' to load the selected PID table.

Figure 5-4: MercuryiTC 'select file' screen for loading alternative PID tables.

The PID values given in the test results for the system are suitable to give good stability. If you wish to improve the stability further, you may be able to do this by adjusting the three terms slightly. In Manual mode, individual PID values can be changed during operation. Control theory and the procedure for optimising the PID values are described in the MercuryiTC manual.

Tapping 'Close' on the 'PID table' screen will return the MercuryiTC to the 'Control' screen. Then tapping 'Home' will return the MercuryiTC back to the home screen.

5.5 Controlling at a set temperature

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. However, the flow can be optimised later.

On the MercuryiTC home screen (see above), tap 'Control'. Select the desired temperature on the MercuryiTC and switch the MercuryiTC heater control to 'Auto'.

It is not necessary to cool the cryostat to base temperature before you set the required 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 hold it at this point. The nitrogen flow can then be optimised so that the heater output on the temperature controller is not too high. In general, the flow should be reduced until the steady heater output is less than 12 V.

5.6 Operation above room temperature

To operate at temperatures exceeding 300 K, the OVC should be continuously pumped on using the OVC evacuation port. Pumping on the OVC continuously will help maintain the high vacuum required to operate the system. Additionally, the needle valve should be fully closed for every operation above 300K.

MAXIMUM SET TEMPERATURE Do not set a temperature higher than 500 K.

After setting up continuous pumping, you may set a temperature exceeding 300 K, using the process described in Section 5.5.

5.7 Changing the sample

A sample within the Optistat DN-V can only be changed by warming the whole cryostat to room temperature, as described in Section 5.8. Then venting cryostat and removing the OVC to gain access to the sample space, as described in Section 5.9.

5.8 Warming up the system

If you do not need to warm the system quickly, it may be left to warm up naturally. To speed up the warming process, pour any remaining liquid nitrogen out of the reservoir and set a temperature of 300 K on the MercuryiTC. To further hasten the warming of the system, allow a small volume of dry nitrogen gas from a bladder into the OVC to break the vacuum.

> **!** BREAKING VACUUM
> When breaking the OVC vacuum, do not use a bladder that has previously been used with helium. Never allow helium gas into the OVC, as it is difficult to pump out again.

5.9 Removing and refitting the OVC

The cryostat OVC needs to be removed from the system if you wish to:

- Change the sample.
- Change the sample holder.
- Repair any mechanical damage.
- Repair the wiring.

Before beginning, ensure that the cryostat has been warmed to room temperature by following the procedure described in Section 5.8. Then, vent the OVC with a dry nitrogen source through the evacuation valve to bring it up to atmospheric pressure.

To remove the OVC, remove the four bolts which hold the OVC to the cryostat top plate. This will allow the OVC to be removed from the rest of the cryostat.

> **!** OVC REMOVAL
> When removing the OVC, ensure the assemblies remain concentric and parallel to each other until they are fully separated. Accidental tilting may cause mechanical damage to the cryostat.

The OVC has now been removed and the desired operations can be carried out.

Before re-assembling the OVC, check the O-ring is clean, undamaged, and lightly greased. Additionally, ensure that the O-ring is firmly pushed into place so it will form a vacuum-tight seal. To reassemble the OVC, ensure it is in the desired orientation and carefully lower the cryostat into the OVC. Secure the OVC to the rest of the cryostat by the replacing the four retaining bolts.

OVC REPLACEMENT

When replacing the OVC, ensure no internal wiring from the cryostat touches the OVC. Such a touch will reduce the performance of the system.

After removing the system's OVC, ensure you recondition the sorb before cooling the system again.

5.10 Changing sample holders

To change the sample holder affixed to the sample stage, the cryostat OVC must first be removed, this can be done following the procedure described in Section 5.9. After the OVC has been removed, there will be clear access to the system sample stage and holder. To remove a sample holder, undo and remove the three M3 retaining bolts, as shown in Figure 5-5. When removing the final bolt, support the sample holder so it does not fall.

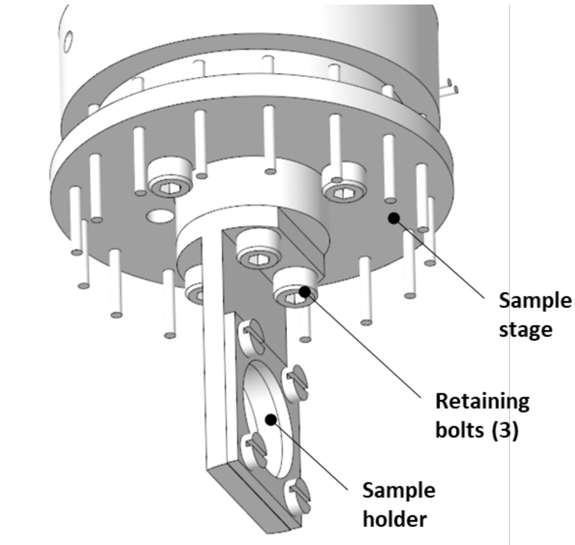

Figure 5-5: Optistat DN-V sample stage.

To refit a sample holder to the system, use the three previously removed M3 bolts affix it to the sample stage. Note that as the sample holder is secured in place with three equally spaced bolts meaning it can be unintentionally affixed in the wrong orientation. Therefore, it is a good idea to check the orientation of the sample holder at this point.

The sample holder has now been changed and the OVC can now been refitted by following the procedure described in Section 5.9.

5.11 Changing system windows

The windows of the Optistat DN-V can be removed so that they can be cleaned or replaced. To change an OVC window, the Optistat DN-V must first be warmed to room temperature and vented with dry nitrogen gas, as previously described in Section 5.8. Each Optistat DN-V OVC window is held in place by four nylon retaining screws, as shown in Figure 5-6.

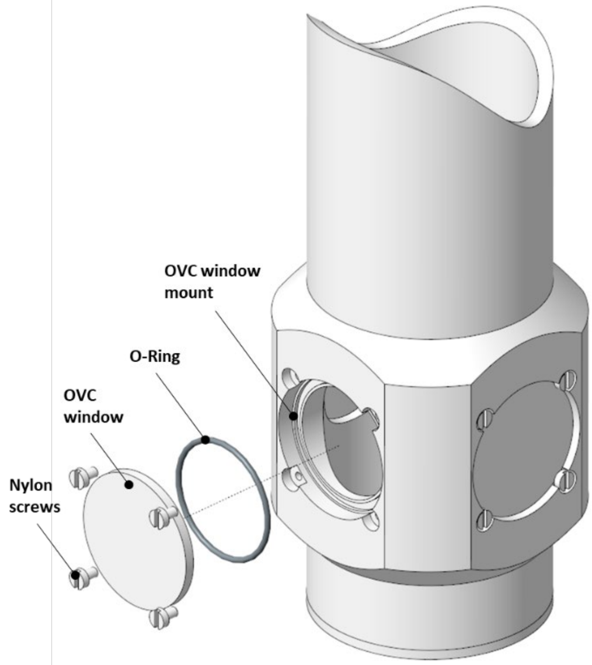

Figure 5-6: Changing the OVC windows.

To replace an OVC window, carefully remove the four retaining nylon screws around the edge of the window, taking care to ensure the window does not fall out by itself when removing the screws. The window can then be removed. Select the new window or blank and clean it using a suitable lens cleaner and lens tissue. Inspect the OVC window mount and O-ring to ensure they are not damaged or contaminated with any debris that could cause an air leak. If required, clean the O-ring with isopropanol and apply a light coating of vacuum grease before re-fitting the O-ring. After replacing the O-ring in its groove, position the cleaned window into the OVC window mount and replace the four retaining nylon screws to secure it in place.

!	OVER-TIGHTENING Do not over-tighten the OVC window screws. Over-tightening could lead to window fracture or poor vacuum sealing.

6 Service and maintenance

The Optistat DN-V will deliver repeatable and reliable performance if maintained properly. This section contains basic and essential maintenance information.

6.1 O-rings

Oxford Instruments recommends replacing the cryostat's O-rings on a two-year cycle. Whenever a part of the cryostat is removed, or if there is a suspected leak on the system, check the relevant O-rings. Ensure that the O-rings are clean, undamaged, and lightly greased. Any damaged O-rings should be replaced immediately.

6.2 Reconditioning the sorb

The OVC will need to be pumped regularly and the sorb should be reconditioned each time the system is cooled down. The procedure for reconditioned the sorb is detailed in Section 4.6.

6.3 Window replacement

Should you need to clean the windows, they can be removed using the procedures described in Section 5.11. Before removing any windows, ensure the system is warm and the OVC vented with dry nitrogen.

6.4 Troubleshooting

Should you encounter a problem with your system, it is first important to establish the source. This could be with the MercuryiTC or within the cryostat itself.

Diagnosis of MercuryiTC temperature controller faults should be made using the MercuryiTC manual T*roubleshooting* chapter. Diagnosis of the transfer siphon should be made using its respective manual. Refer to the troubleshooting recommendations in Table 6-1 for problems arising from the cryostat itself, or a combination of the above.

If you are unable to resolve the problem, please direct all enquiries through your nearest support facility. Please provide a full set of test data for diagnosis, along with details of any additions or modifications that you may have made to the system.

Issue	Possible cause	Recommendation
Poor temperature control	Incorrect temperature controller PID settings	Refer to the system's test results and the MercuryiTC manual.
Heater or sensor wiring fault	Wiring short or break	Check wiring resistances and compare with values in the factory test results as in Section 8.1.
Poor cryostat OVC vacuum	Leak in to vacuum space	Examine O-rings for contamination or damage. Check windows and electrical connectors are correctly sealed and undamaged. Check pressure relief valve is sealing correctly. Use a leak detector to identify the leak.
	Water contamination	Warm up thoroughly to ensure all internal surfaces are free of condensed water. Additionally, pump the outer vacuum chamber with a rotary pump, with gas ballast valve open.

Issue	Possible cause	Recommendation
Poor cryostat OVC vacuum	Sorb contaminated	Recondition the system sorb as per the procedure given in Section 4.6.
High base temperature	Heater still on	Check the heater is switched off.
	Poor vacuum	Check the quality of the vacuum in the cryostat.
	Sensor fault	Check sensor resistance at base temperature and compare with the supplied sensor calibration data. Remember to account for the resistance of the wiring.
	Wiring heat load	Check that too much heavy wiring has not been added to the sample holder, introducing a high heat load.
	Exhaust blockage	Check whether there is any flow of gas from the exhaust valve. If the sample space capillary is blocked, warm it up and blow dry gas through it to remove moisture.
Condensation or frost on OVC	Poor cryostat vacuum	Check poor cryostat vacuum actions.
Sample cannot be warmed up	Low set temperature	Check the set temperature is higher than the present sample temperature or switch the heater on manually.
	Heater wiring fault	Check that the heater circuit is not open or shorted by checking the resistance between pins A and B on the diagnostic connector, checking for isolation between these pins and ground.
	Low heater voltage limit	Check that the heater voltage limit on the temperature controller is high enough. Normal settings are limited to: 40 V, Resistance: 20 Ω.

Table 6-1: Common issues and most likely causes.

7 Optistat DN-V specifications

7.1 Performance

Performance	Specification
Temperature control range	77 K† - 500 K
Temperature control stability	± 0.1 K
Liquid nitrogen hold time (at 77 K)	15 hours
System cooldown time	20 minutes

7.2 Electrical power

Component	Power Consumption	Voltage	Frequency
MercuryiTC temperature controller	450 W	100 - 240 VAC	50 - 60 Hz

7.3 Physical

Parameter	Nominal Value
Weight (kg)	5
Dimensions W x D x H (mm)	175 x 175 x 450

7.4 Technical exclusions and assumptions

Performance specifications are given for standard configurations and intended use. Siting, environment, system variations, modifications and upgrades may affect the performance.

- The standard cryostat is fitted with four optical radial windows and one blank axial window.
- † Base temperature will depend on the boiling point of liquid nitrogen which is impacted by atmospheric pressure.

8 Appendices

8.1 Checking the wiring

A resistance meter can be used to check the diagnostic wiring on the cryostat. You should expect to measure the following values.

Pins	Approx. resistance (at 300K)
A - B	15 – 25 Ω
C - D	110 – 120 Ω
C - E	3 – 6 Ω
C - F	110 – 120 Ω
D - F	3 – 6 Ω
K - L	145 – 155 Ω
A - C	> 1 MΩ
A - ground	> 1 MΩ
C - ground	> 1 MΩ

Table 8-1: Expected resistance measurements for 10-pin cryostat connector.

8.2 Cleaning and general care

All stainless-steel surfaces may be cleaned with water or IPA (Isopropanol Alcohol), a mild abrasive may also be used like “scotchbrite” on matt or unpolished surfaces.

All painted surfaces and labels should be cleaned with warm soap and water, no solvents or abrasives should be used.

CLEANING SOLVENTS

Never use incompatible solvents when cleaning. Never clean the system when cooling, cold or evacuating as this may lead to an O-ring failure and may result in vacuum loss which could damage the system.