Operator's Handbook

HelioxVT

Insert Data

Issue 2

August 2005

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Contents

Warnings

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

1 Details of your Heliox insert

1.1 Introduction

The standard manual for the Heliox explains how to set up and run the insert. However, most of these inserts have some special features, and details of these features are given in this booklet.

1.2 Specifications of the insert, and special features

Base temperature		0.3 K
Hold time at base temperature		> 40 hours
Temperature with 50 μW heat load		< 0.35 K
Hold time with 50 μW heat load		> 8 hours
Temperature control	- in range 0.28 to 1.2 K	± 3 mK expected
	- in range 1.2 to 300 K	± 0.1 K
Sample length - without magnet		See GA
Nominal weight (without magnet)		7 kg

1.3 Experimental wiring

The experimental wiring is fitted in the IVC pumping line, and it is terminated on the connector below the 1 K pot. All wires are constantan as standard.

Additional wiring may be fitted in the spare ports.

1.4 The ITC temperature controller

It is recommended that an ITC temperature controller is used to control the Heliox system. Several range cards have been developed to customise this controller for use with Oxford Instruments ^{3}He systems.

The ITC502 or ITC503 may be used to monitor and control the temperature of the sorb and sample, over the entire operating range of the system. It can also be used to drive an auto needle valve. If you use two ITC temperature controllers you can achieve better control in the high temperature range and drive a second auto needle valve stepper motor.

The ITC is set up with the following range cards.

Temperature controller model: ITC503

Card No.	Type of Sensor	Temperature range (K)
1	Allen Bradley, 100 ohm	1.5 - 250
2	RuO_2 resistor, 2210 ohm	0.250 to 9.999
3	Cernox 1050 AA	1.0 to 300.0

The software provided with the ITC502 or ITC503 allows the ranges to be altered easily to suit a wide range of sensors. Internal switches may need to be altered to change the excitation current and other parameters. The ITC manual contains full details of this procedure.

1.5 Requirements for commissioning the system

The following equipment will be needed for the successful commissioning of a Heliox system. Any equipment which has been supplied with the system will be marked with a ✓on the left margin.

1.5.1.1 Pumping system requirements

Pumping system for the 1 K pot, (including a single (or double) stage rotary pump with a displacement of 8 m^3/hour and the valves and interconnecting lines as shown in the manual). This pump may also be used to evacuate the IVC.

A mass spectrometer leak detector system is not required for routine operation of the system, but access to one may be useful if a leak is suspected.

1.5.1.2 Electronic accessories.

A multimeter for general testing of control and experimental wiring, with volts, amps and ohms ranges.

Either an Oxford Instruments ITC temperature controller, or an equivalent instrument for monitoring the temperature of the sorb, supplying power to the sorb heater, and (if required), controlling the sorb temperature to maintain a steady sample temperature.

Either an ITC temperature controller or an equivalent instrument for monitoring the temperature of the sample, and controlling it, (if required).

If very accurate thermometry is required it is necessary to obtain the relevant equipment to monitor the thermometer.

1.5.1.3 Other accessories

Suitable cryostat (with clear neck diameter at least 50 mm)

1.5.1.4 Consumable materials and electrical power requirements.

Liquid helium and liquid nitrogen

2 Test results

Base Temperature, measured with calibrated thermometer	0.240	K
Hold time at base temperature	48	hours
1K plate temperature when insert running at base	1.5	K
Hold time with 50 μW heat load	10.5	minutes
Measured temperature with 50 μW heat load	0.295	K
VTI helium boil off (with insert at base temperature) in continuous fill mode.	180	cm^3/hour
VTI temperature when insert running at base	1.48	K

Note: for helium, 1 litre/minute NTP gas ≡ 90cm^3/hour liquid at normal boiling point

Typical condensation parameters

SORB temperature	45	K
1K plate temperature	1.89	K

High temperature sensor calibration

Sensor serial number X35860

Temperature control in high temperature range

Temperature (K)	Stability (mK)	1 K pot sensor (K)	1 K pot Flow (l/min of gas at NTP)	Sorb temperature (K)	P	I	D
5	±20	2.38	3.5	23.98	10	2	0
280	±100	5.428	14	5.614	10	2	0.6

Heater voltage limit (high temperature range) 40 volts

Temperature control in low temperature range

Temperature (K)	Stability (mK)	P	I	D	Sorb temperature (K)
0.4	± 1	10	2	0	12.57
0.9	± 2	10	2	0	19.42
1.2	± 3	10	2	0	20.44

* Using card 2 to control (others using card 3).

Heater voltage limit (low temperature range) 40 volts

Operator's Handbook

HelioxVT

Sorption pumped ^{3}He Insert

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OXFORD INSTRUMENTS

Contents

Warnings

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

Other information supplied with this manual

The following information is supplied with this manual or available on request:

- Safety Matters - essential information to help you to run a system safely
- Practical Cryogenics
- Heliox insert data sheet
- Thermometry and resistor calibrations for Heliox systems
- Useful reference books

1 Introduction and description

This manual is designed to introduce you to this sorption pumped ^{3}He refrigerator, whether you are experienced in the operation of cryogenic systems or a beginner. It is a relatively simple ^{3}He system to run because the ^{3}He gas is safely stored within the Heliox insert at all times.

In you have bought a complete system from Oxford Instruments, a separate system manual will have been supplied. You should cool down the system as described in the system manual before you load the Heliox insert in the Variable Temperature Insert (VTI). This free standing manual then describes how to load the Heliox insert into a cold system.

1.1 Safety

Please refer to the separate booklet, "Safety Matters". This includes information about the properties of liquid nitrogen and liquid helium, and detailed recommendations about the precautions that you should take. It is your responsibility to ensure your own safety, and the safety of people working around you.

1.2 General description of the complete system

The HelioxVT system is a low cost, short lead time ^{3}He refrigerator designed to operate in an Oxford Instruments' Variable Temperature Insert (VTI). The VTI may form part of a superconducting magnet system, or part of a stand alone cryostat (an Oxford Instruments' Variox, for example). The sample is changed by warming the entire insert to room temperature, and removing the IVC. The Heliox is cooled to <2 K by the VTI through exchange gas. The Heliox will run in any VTI that it fits in to, but only a VTI/Heliox system supplied by Oxford Instruments will guarantee the optimum performance from the Heliox.

A leak detector or turbo pump is required to pump exchange gas from the IVC of the Heliox.

At temperatures below the base temperature of the VTI, the temperature of the sample and the sorption pump are controlled by an Oxford Instruments ITC502 or ITC503 temperature controller (if ordered). It is possible to set up a computer interface to the ITC using IEEE or RS232. This allows you to control the operation of the insert automatically using Oxford Instruments ObjectBench or B-T environment software. Above the base temperature of the VTI, the temperature of the sample is controlled through the temperature of the VTI.

The operating temperature range of the insert is from 0.3 K to 300 K, but the upper limit of the temperature range might be set by the working range of the VTI.

A range of calibrated thermometers is available for the Heliox insert to help you to measure the temperature of your sample accurately. The insert is fitted with uncalibrated thermometers for the range below 1 K as standard. The high temperature sensor is calibrated as follows:

- RhFe resistors have a three point calibration
- Carbon glass resistors have a 30 point calibration
- Cernox resostors have a 39 point calibration

1.3 Brief description of the Heliox insert

Refer to the drawings of the Heliox in this manual.

The insert has a vacuum seal which allows it to be loaded into the VTI without allowing contamination of the neck of the VTI with ice. The inner vacuum chamber (IVC) is sealed by a greased cone seal, allowing the system to be used by relatively inexperienced personnel.

Caution: Anyone working with the Heliox Insert must be familiar with the precautions they must take to ensure their own safety and the safety of those people working around them.

There is no need to make indium seals during the sample changing procedure. There are no indium seals on the insert at all.

The spare port gives line of sight access through the insert from room temperature to the sample space. You can use it to install services for your experiment. However, it is important to heat sink all of these services effectively to minimise the effect on the insert's performance.

The charge of ^{3}He is sealed into a self contained storage vessel so that it is not necessary to remove the valuable gas from the insert when it is warmed to room temperature. This reduces the complexity of operation of the system. The (nominal) 2.5 litre charge is stored at a pressure of approximately 2 bar (absolute). The storage vessel is fitted with a pressure relief device.

The sample is mounted in vacuum on the underside of the ^{3}He platform, or on the end of a low eddy current sample holder at the field centre of a superconducting magnet.

1.4 Principle of operation of the Heliox insert

The sample is mounted in vacuum on the base of the ^{3}He pot, or on the sample holder as shown on the drawing inserted in this manual. When the sample has been mounted and the IVC has been sealed and evacuated, the insert may be cooled by lowering it into the VTI.

The insert is designed to allow the temperature of the sample to be controlled in the range from 0.28 to 300 K, and this is done using two separate modes of operation. Below the base temperature of the VTI, typically 1.5K, the Heliox runs as a sorption pumped ^{3}He insert. Above 1.5K the IVC of the Heliox is flooded with exchange gas and it acts as a sample rod in the VTI. Please refer to the manual for the VTI for operation above 1.5K.

1.4.1 Operation as a sorption pumped ^{3}He system

The Heliox consists of three main components, connected by a tube (see Figure 1):

1. The sorption pump (or 'sorb'). This is a volume containing a quantity of charcoal that has a very high surface area. When cold, this charcoal is capable of pumping, at high vacuum, more than 0.1 moles of ^{3}He gas before it becomes 'saturated'. Once the surface area is saturated with ^{3}He, it no longer pumps, and it must be heated ('out-gassed') in order to liberate the ^{3}He gas that it has adsorbed. During condensation, the sorb will be warmed to >30 K for ~1/2 hour, which is enough time for all the ^{3}He to condense.

2. The 1K plate. This plate is designed to remain at the temperature of the VTI (~1.5K) even when the sorb is being out-gassed. It is here that the 3He gas condenses, and then drops down into the 3He pot.

3. The 3He pot. This is a reservoir for the 3He liquid. Once all the 3He has condensed into the 3He pot, the sorb is allowed to cool, and it starts pumping the gas away. As the vapour pressure drops, so does the temperature of the liquid 3He, and the sample attached to the base of the 3He pot cools too. The ultimate base temperature achieved is determined by the heat load on the 3He pot from its surroundings, and the pumping speed of the sorb.

To regulate the temperature above the natural base temperature of the Heliox, the pumping speed of the sorb is reduced by warming it. By heating the sorb and not the ^{3}He pot, the hold time at the desired temperature is not decreased.

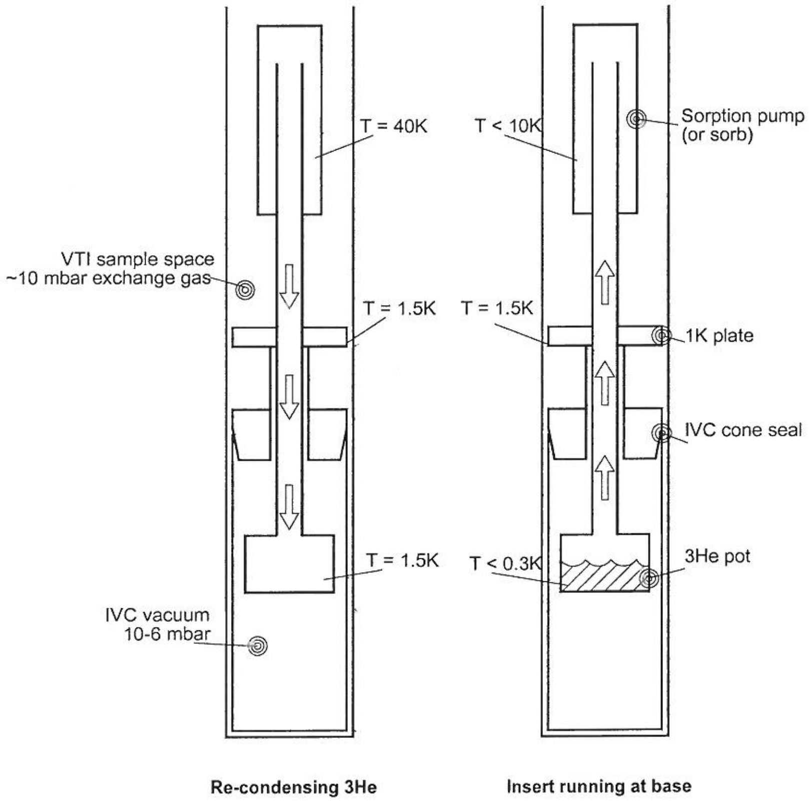


Figure 1 Operation principle of a sorption pumped ^{3}He insert

2 Commissioning and routine maintenance

2.1 Commissioning requirements

The Heliox Insert data booklet lists the typical commissioning requirements and indicates which parts have been supplied with the insert.

2.2 Unpacking and initial assembly of the system

The system should be unpacked carefully and inspected for any damage caused during shipment from OxforInstruments. The IVC should be removed as described in section 3.1, and the packing piece should be removed from the underside of the ^{3}He pot before attempting to run the system.

Check to ensure that none of the components are missing. If any problems are encountered you should contact Oxford Instruments (through the agent if appropriate).

2.3 Control wiring details

2.3.1 Insert control wiring

Ten Pin Fischer connector 1

Pin		Function
A	)	Sorb heater (1 x 34 ohm heater)
B	)	
C	V-	) 4 wire
D	V+	) Sorb (100 ohm Allen Bradley)
E	I+	) Temperature
F	I-	) Sensor
H	V-	)
J	V+	) 1 K plate
K	I+	) sensor (2200 ohm RuO_2)
L	I-	)

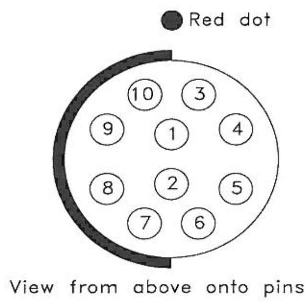


Figure 2 Pin numbers on a hermetically sealed Fischer connector 1031 Z010 (DBEE), viewed from the outside of the cryostat

Ten Pin Fischer connector 2

Pin		Function
A	)	^{3}He pot heater (200 ohm wire wound)
B	)	
C	V-)	High temperature sensor on ^{3}He pot
D	V+)	
E	I+)	
F	I-)	
H	V-)	
J	V+)	Low temperature sensor
K	I+)	on ^{3}He pot (RuO_2)
L	I-)	

2.3.2 Connections to ITC temperature controller

The ITC is configured to read the following sensors:

	Calibration	Range	Sensor location
Channel 1	100 Ω Allen Bradley	1.4 - 250 K	Sorb
Channel 2	2200 Ω RuO_2	200 mK - 7 K	^{3}He pot or 1 K plate
Channel 3	Cernox or RhFe	1.5 - 300 K	^{3}He pot

Cables are supplied which allow connection from the 9-way 'D' connectors on the ITC to the 10 way Fischer connectors on the Heliox. (Please refer to the ITC manual to find more details of the pin connections). These cables also make electrical connection to the heaters associated with each sensor. These heaters are the sorb heater (34 Ω), and the ^{3}He pot heater (200 Ω).

For operation at the lowest temperatures, it is necessary to un-plug the high temperature sensor from the back of the ITC (channel 3), since the excitation current used on this channel dissipates enough heat in the sensor to represent a significant heat load on the ^{3}He pot.

The same type of sensor is used on the ^{3}He pot and the 1 K plate, and the temperature of the 1 K plate can be monitored by the ITC if required. Disconnect the lead from Fischer connector 1, and swap the lead from Fischer connector 2 to connector 1. The approximate temperature of the 1K plate will now be displayed on channel 2. In normal operation, the 1K plate will be at the same temperature as the VTI (or slightly warmer), and using the ITC to measure the 1K plate in this way need only be used to diagnose problems.

2.4 Routine maintenance

It is unlikely that any routine maintenance will be required on the Heliox insert, but some of the accessories may require maintenance at intervals recommended by the manufacturer.

2.4.1 Pumping system

The recommendations of the manufacturer of any vacuum pumps should be followed.

Note: Most vacuum pumps require regular servicing, and the oil must be changed at the intervals specified by the manufacturer.

3 Mounting the sample and final assembly of the system

3.1 Removing the IVC

The greased cone seal cannot easily be removed without using the slide hammer which is supplied with the insert. This is because the cone has a locking taper to ensure that the IVC does not fall off accidentally.

Caution: Do not attempt to remove the IVC by prising it away from the flange or by twisting it relative the insert. Use the slide hammer carefully as described below.

Vent the IVC by opening the valve in the pumping line. Screw the slide hammer fully onto the thread at the bottom end of the IVC, and hold the insert firmly. Slide the moving part of the hammer sharply away from the insert, taking care not to trap your fingers between it and the end flange. When the moving part hits the end flange the impact will tend to pull the IVC tube away from the flange.

Caution: It is important that you operate the hammer gently to avoid straining the vacuum tight soldered joints on the IVC. It should take between 5 and 10 gentle operations to remove the IVC.

3.2 Experimental access on the insert

The experimental wiring on the insert is described briefly in the separate Heliox insert data booklet. You can add extra wiring to the insert to suit your experiment. It is important to realise that the number of wires connected to the sample may affect the low temperature performance. In particular, it is important to consider the amount of heat conducted down the wires; in general it is preferable to use thin high resistance wires whenever possible. A heat load of 10 to 15 μW may reduce the hold time of the insert by a factor of two.

The spare port may be used to bring other services down to the sample space. Take care to ensure that additional services are heat sunk effectively, and that the lower end of the spare port tube is covered with foil to minimise thermal radiation leaks into the IVC.

3.3 Mounting the sample

Remove the IVC as described in section 3.1.

The sample is mounted in vacuum on the base of the ^{3}He pot. Several tapped holes are provided for this purpose. The sample must be thermally anchored to the ^{3}He pot if it is to be cooled efficiently. In general the best thermal contact is achieved by metal to metal contact between two clean copper surfaces firmly pressed together. If preferred, the copper surfaces may be gold plated to prevent corrosion.

If the sample is delicate and non metallic, it is often sufficient to cool it through the electrical leads, which should be thermally anchored to the ^{3}He pot.

Connect the wiring to the sample, and check all the wiring carefully before you fit the IVC.

3.4 Fitting the IVC using the greased cone seal

The IVC is sealed onto the insert using a greased cone seal. This type of seal is quick and simple to make, and it requires much less cleaning time than an indium seal when the IVC is removed again.

Caution: It is important to ensure that the surfaces of the cone seal are kept clean and free from scratches. It is good practice to remove the grease from the mating surfaces as soon as the IVC is removed so that dirt is not allowed to collect on them.

Make sure that your hands are clean so that no small particles are allowed into the seal. The seal is made using ordinary silicon based vacuum grease. Rub a thick layer of grease thoroughly onto the surface of the cone on the IVC flange; rub a thin uniform layer onto the inner surface of the IVC tube. Fit the IVC carefully over the insert, making sure that none of the grease is removed by the ^{3}He pot, 1 K plate or the sorb. Push the cone into the IVC with a slight twisting action.

Evacuate the IVC to a rough vacuum (<1 mbar). This will pull the cone seal together very firmly, and it will not be possible to separate it again without using the slide hammer provided with the insert. The cone is designed to be a locking taper, so that the IVC will not fall off accidentally even if it is vented to atmospheric pressure.

3.5 Preparation for cool down

After you have pumped the IVC to a rough vacuum, close the IVC valve and remove the pump. Allow a small amount of (recovery grade) helium gas into the IVC. Approximately 10 cm^3 (NTP) should be sufficient. The best method for allowing a small amount of helium to enter the IVC is a bladder fitted to the IVC valve.

4 Running the Heliox insert

4.1 Cooling the insert down

Prepare the insert as described in section 3. The insert is now ready to be inserted into the VTI. This operation ahould take approximately 30 to 40 minutes once you are familiar with the proceedure. Connect the insert wiring to the ITC temperature controller so that the cooldown can be monitored easily. If the ITC is connected to a computer, the temperatures can be logged to show the rate of cooling.

Warning: **Make sure that you are aware of the precautions that you must take to ensure your own safety, and the safety of other people working around you. You should receive proper training from a competent person before using cryogens.**

4.1.1 Pre-cooling using liquid nitrogen

The most rapid cooldown can be achieved using a deep bucket of liquid nitrogen to pre-cool the insert. Lower the insert slowly into the liquid nitrogen, and support it so that it will not fall over. The time required for pre-cooling will depend to some extent on the mass of the sample. If the sample is very light, the ^{3}He pot should reach 100 K after approximately 15 to 20 minutes.

Caution: Pre-cooling in liquid nitrogen should not be employed if the IVC is of bi-metallic constuction (eg. Aluminium/Beryllium, or Aluminium/Brass), since the differential thermal contraction may break the joints between the metals if they are cooled too quickly.

4.1.2 Loading the insert into the VTI

The Heliox insert can be treated like any sample rod for the VTI. A full description of the procedure for changing sample rods can be found in the manual for the VTI. A brief description is included here:

The sample space of the VTI should be pressurised to slightly more than one atmosphere of helium to avoid contamination being 'sucked in' during changes of the sample rod. The needle valve on the VTI should be shut. Remove the blanking cap (or the previous sample rod) from the VTI and insert the Heliox.

Start to pump the VTI. Open the needle valve on the VTI gradually, to set sufficient flow of helium through it. If a pressure gauge is available on the VTI pumping line, then the needle valve should be adjusted to maintain a pressure of 50-100 mbar. As the VTI cools, the flow of gas will increase (especially once it is below 50 K) and the needle valve will need to be closed gradually.

If the insert is to be used at a temperature below 1.5 K the procedure in section 4.2 should now be followed. If it is to be used only at higher temperatures, follow the procedure in manual for the VTI to control the sample temperature.

4.2 Pumping exchange gas, 3He condensation, and cooling to base temperature

When the VTI reaches ~10 K, the exchange gas should be pumped from the IVC using a leak detector or turbo pump. Connect the pump to the NW16 fitting on the insert, pump down the lines, and open the vacuum valve on the insert. If a leak detector is used, wait until the signal is <10^{-5} mbar litres/sec. If a turbo pump is used, it is usually sufficient to pump for 1 hour.

Note: It is important that the IVC is pumped while the VTI temperature remains higher than 4 K. If the VTI cools to below 4K during pumping, the temperature of the VTI should then be set to 4 K. Once the exchange gas has been pumped, the VTI should cool to its base temperature.

It is possible to control the Heliox insert automatically from a computer using Oxford Instruments B-T environment or ObjectBench software. The software manuals explain how to do this.

When the insert is precooled to below 6 K as described above, it is ready for condensation of the ^{3}He gas.

Set the sorb temperature to 30-40 K to ensure that it will not pump the ^{3}He gas. (Take care to ensure that the correct heater (1) is selected on the ITC so that the sample and ^{3}He pot are not accidentally warmed). As the 1 K plate cools below 3 K, the ^{3}He gas will start to condense and run down into the ^{3}He pot. The sample temperature will be seen to drop quickly to around 1.5 K. Once the ^{3}He pot and the 1K plate have reached a stable temperature, condensation is complete. The condensation process should take between 20 and 30 minutes. This method of condensation is sufficient for normal operation and it is used by the software for automatic control of the insert

When the sample temperature is steady, the insert can be cooled to base temperature. This is done by cooling the sorb, switch off the sorb heater by setting the sorb temperature on the ITC to 0 K. The sorb will be found to cool over a period of minutes and the sample temperature should drop with the sorb temperature. If the sample is light and has a high thermal conductivity, it should reach base temperature within 15 to 25 minutes.

If the experimental heat load is very low, the insert should stay at base temperature for longer than 30 hours. The VTI temperature must be maintained at less than 2 K while the Heliox is at base temperature. The ITC that controls the VTI (if one was ordered) will automatically control the needle valve to ensure that the VTI does not run out of liquid helium (refer to the manual for the VTI).

The base temperature and hold time of the insert may be slightly affected by the self heating of the high temperature range sensor. If the lowest possible temperature and longest possible hold time are desired, it is necessary to unplug the high temperature sensor from the ITC (channel 3). This will ensure that the excitation current in the sensor does not supply heat to the system. The base temperature may be improved by 10 to 30 mK, and the hold time may be doubled by disconnecting the sensor.

4.3 Temperature control

The following instructions are intended to help you to understand the factors affecting the temperature of the sample. The test results for the insert will allow you to control the insert to the level required by the specifications. Settings to achieve improved stability may be found with some experience.

In the low temperature range the sample is usually monitored using a RuO_2 resistor mounted on the ^3He pot. These resistors are quite insensitive to a magnetic field and so are very useful for control of temperature, even when the field is swept. If the temperature is to be measured accurately, it is recommended that a calibrated sensor is used; for example, a germanium or RuO_2 resistor.

Two separate heater outputs from the ITC are required (for example, for sorb control and for high temperature control using the ^3He pot heater), so a switching card (part number CQB 1800) is fitted to the back of the instrument to switch the heater output automatically. The switching card should be fitted to the ITC and the link between the heater outputs from card 1 and card 2 (marked LK 1-2 on the printed circuit board) should be made by soldering a zero ohm resistor onto the board. The switching card is not needed if the insert will only be used at temperatures below 1.5 K, but the heater on the ^3He pot must then be disconnected. As an option to the CQB1800 a CQB2200 heater share box can also be supplied.

The level of temperature control can be improved over the entire range by adjusting the maximum heater output available from the ITC temperature controller. It is best to balance the maximum rate at which the temperature can rise or fall when it is outside the proportional band. For example, if the sample or sorb temperature can rise twice as quickly as it can fall, it may be helpful to reduce the maximum heater output by a factor of two. This can be optimised by plotting the temperature against time and matching the gradients.

Card 1	For use with the sensor fitted to the sorb (for example, Allen Bradley carbon resistor). This gives an indication of the temperature of the sorb during re-condensation of the ^3He. Note that the accuracy of this sensor is not critical for successful operation of the system.
Card 2	For measurement of the ^3He pot temperature with resolution of 1 mK. The card uses a RuO_2 resistor as its temperature sensor.
Card 3	For measurement of the ^3He pot temperature in the range 1.5 to 300 K. The card typically uses a Cernox resistor.

Table 1 ITC Range Cards (A summary of the available options).

4.3.1 Temperature control in the low temperature range

a) Basic Control (background information)

Coarse control can be achieved by setting the temperature of the sorb to a constant value and thereby setting its pumping speed. If the sorb is below 8 K, so the ^3He temperature will be reduced to a minimum for a given experimental heat load.

If the sorb is above 40 K, it will not pump, and the ^{3}He temperature will be largely dependent upon the temperature of the 1 K plate. Intermediate sorb temperatures are used to control the ^{3}He temperature within these limits.

When the temperature of the sorb is controlled at a constant level by an ITC temperature controller, the sample temperature tends to drift up slowly as the charcoal saturates. If there is no experimental heat load, it should be expected to rise by about 0.01 K per hour.

The upper temperature limit for this method of control is the temperature of the 1 K plate. That is, at some stage further warming of the sorb will not result in further warming of the sample because these are the conditions used during the condensing procedure.

b) Temperature control with feedback from the sample temperature sensor
The level of control can be greatly improved by measuring the sample temperature with the ITC, and arranging for the controller to adjust the temperature of the sorb continuously to maintain a steady sample temperature. In this way, the temperature can be held stable within a few milli-kelvin. In order to achieve this, an extra range card is used, and this is described in Table 1 (see card 2).

The settings on the three term controller of the ITC (that is, PID settings) need to be optimised to suit the operating conditions. The test results will indicate the settings used at Oxford Instruments, and should be regarded as a starting point. Improved stability may be achieved by fine adjustment. It is very difficult to optimise the settings in a region where the cooling and warming effects are not balanced. For example, it is difficult to control accurately below say 0.32 K, because warming the sorb slightly has a large effect on the ^{3}He temperature and because the time constant for reduction in temperature is quite long.

If a magnetic field is swept very rapidly the temperature stability may be affected because of the eddy current heating in the metallic components in the sample region.

Warning: **Set the maximum temperature limit of the sorb (card 1) to approximately 80 K. The highest ^{3}He temperature that can be achieved is the temperature of the 1 K plate. Further warming of the sorb has little effect. Therefore it is possible for the sorb to continue to warm in an attempt to warm the sample further, and thermal runaway may occur. If this temperature is reached, the heater will be turned off, and there will be no risk of damage to the wiring and heaters. The ITC manual describes how to set this maximum temperature.**

4.3.2 Temperature control at higher temperatures

A Cernox or RhFe sensor is used as the high temperature sensor as standard; the choice depending on the magnetic field of the sample environment.

The simplest way to control at higher temperatures is to flood the IVC with exchange gas and set the temperature using the VTI. This method should work over the whole temperature range of the VTI (1.5 - 300 K), but the temperature of the ^{3}He pot may be slow to respond to changes in the VTI temperature.

Alternatively, temperatures above that of the 1 K plate can be achieved by supplying heat directly to the heater on the ^{3}He pot. Keep the IVC under high vacuum. In this mode of temperature control, the sample temperature is controlled by balancing the heat supplied on the ^{3}He pot with the cooling power of the insert. The sample is cooled by conduction through, and convection of, the ^{3}He gas in the central tube of the insert. In order to keep a reasonable pressure of ^{3}He gas in the central tube, it is necessary to apply some heat to the sorb. The temperature of the sorb is not critical but ideally it should be kept within the temperature range from 30 K to 50 K. (This is handled automatically if a heater share box CQB2200 is used, if this box is not available, a separate power supply needs to be found). Select channel 3 for high temperature control of the insert.

The three term controller settings for the ITC will be found to be significantly different from those used in the low temperature range, and may vary over the high temperature range as indicated in the test results. The test results will indicate the settings used at Oxford Instruments, and should be regarded as a starting point to allow you to control to the specifications. Better settings may be found by trial and error.

This type of insert is always found to cool samples quite slowly in the high temperature range. If you want to take readings while the temperature sweeps rapidly make sure that you take the lowest temperature readings first.

4.3.3 Autotuning temperature control with the ITC503

The software supplied with the ITC503 includes a facility to help determine the optimum temperature control settings (the PID settings). This facility is described in the software manual.

Temperature control within the specifications of the insert can usually be achieved by using the autotune facility with the full heater output voltage and reasonably coarse autotune conditions. A step size of 0.1 K and overshoot of 20% in the low temperature range, and a step size of up to 5 K and overshoot of 10% in the high temperature range should give reasonable values for P, I and D after approximately 30 minutes per temperature.

The most accurate PID settings are found when the rate of warming with full heater power is approximately equal to the rate of cooling with zero heater power. In general, this condition can be achieved by adjusting the maximum heater output voltage. Typical values for a Heliox insert are between 5 and 10 volts in the low temperature region and around 40 volts in the high temperature region, although these will vary with the experimental heat load and the sorb heat exchanger flow.

Once PID settings have been found for a temperature range, they can be stored in a "look up" table. The ITC503 will use PID settings from the "look up" table if it is switched to the "Auto PID" mode.

4.4 Warming the system to room temperature

The procedure to be followed to warm the insert to room temperature is quite straightforward, and it can be done quickly (typically 10 to 15 minutes). However it is important to remember several points for safety reasons.

Warning: **When you warm up the insert make sure that the IVC is free to vent safely. Any remaining cryogens or contamination can then escape without causing damage to the insert.**

Connect an empty bladder to the IVC pumping line and open the valve. This ensures that any gas that has leaked into the IVC can expand freely into the bladder. It is unlikely that the bladder will expand at all as the insert is warmed since there should be no gas in the IVC.

Shut the needle valve on the VTI and set the VTI temperature to >100K. When the sorb sensor on the Heliox reads >100 K, the insert can then be pulled out of the dewar quickly. If the Heliox has not been allowed to warm above 100K, some liquid air will run from the cold metal as it is withdrawn, so it is preferable to lift the insert out quickly to prevent the collection of solidified air in the VTI. Cover the top of the VTI as soon as possible, using a blank flange or bung.

The insert may now be warmed gently with a hot air blower. If the IVC was under high vacuum, some exchange gas should be re-introduced. It is important to ensure that the IVC is not removed before the ^{3}He pot is warmed to near room temperature to prevent the condensation of moisture on the sample.

The IVC may then be removed using the slide hammer as described in section 3.1.

4.5 Fault Finding

This section begins with a more detailed explanation of the workings of the heliox, in order to clarify the possible causes of a fault. Particular faults are then discussed with respect to the correct functioning of the heliox. If the problem cannot be solved with the aid of this manual and another operational fault is suspected, a full set of test data should be produced and sent to Oxford Instruments for diagnosis along with details of any additions or modifications that you have made to the system.

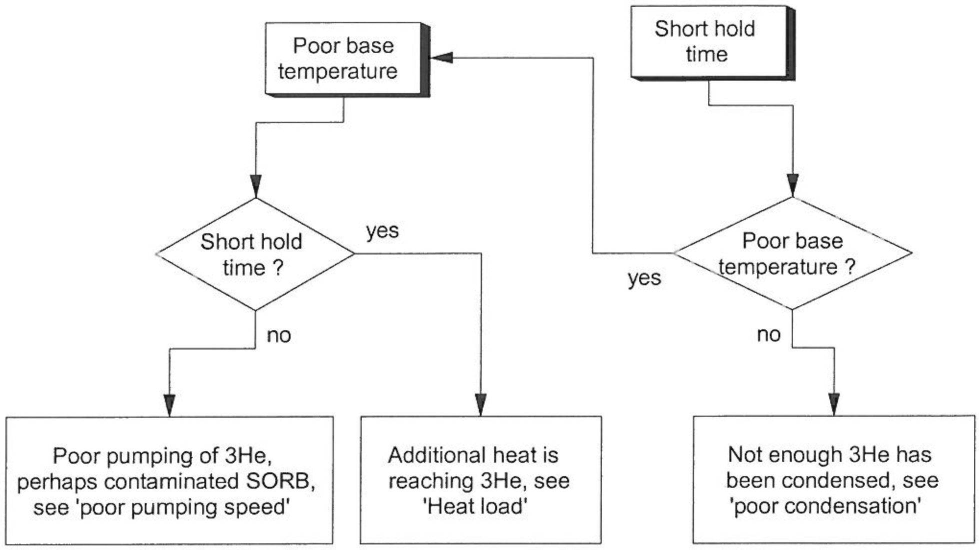
4.5.1 Normal operation

After condensation, there will be n moles of 3He liquid sitting in the 3He pot. Once the sorb has been pumping for a while, and the 3He has cooled to base temperature, a steady state is established where the 3He flows up to the SORB at a rate of $\dot{n}$ moles/sec. The hold time of the system is then given by $n/\dot{n}$. The heat load on the 3He pot is:

$$\dot{Q} = \dot{n}L$$

where L is the latent heat of 3He at base temperature (26.2 joule/mol @ 300mK, varying slowly with temperature). $\dot{Q}$ is the total heat load on the 3He pot from all external sources, which may be through the support and wires connected to the pot, or through heat applied by the user. When the Heliox is running at base temperature, and no heat is being applied by the user, the remaining heat load is called the heat leak, $\dot{Q} = \dot{Q}_{LEAK}$.

In general, poor performance of the heliox can be attributed to one of three causes. The following flow diagram explains how to distinguish between these three general problems, then they are each explained in more detail:



4.5.2 High heat load

This means that $\dot{Q}_{LEAK}$ is too high. This causes $\dot{n}$ to be too high, and the hold time at base temperature will be too short. In addition, the base temperature is degraded because the pressure (and hence temperature) in the 3He pot must be high in order to drive the high flow rate. This is probably the most frequently encountered problem with HelioxVT inserts, possible causes are

- Temperature of 1K plate too high - compare the temperature of the 1K plate with that achieved during system test. If this is the case then check the temperature of the VTI, and adjust the flow so that the 1K plate cools.

Note: If the Heliox is being used in a different VTI from the one it was tested in it may be impossible to reach the same temperature on the 1K plate.

- High level of exchange gas in IVC. Check the quantity of 4He gas in the IVC with a leak detector, this should be give a leak rate signal <10^{-5} mbar litre/sec. The cone seal is the easiest joint to check; see section 4.2.

- Too much or too heavy wiring has been fitted to the insert and is conducting too much heat into the ^{3}He. Try to improve the heat sinking, reduce the diameter of the wiring or increase its length.

- One of the ports on the IVC is not baffled off sufficiently, and heat is being radiated from the warmer regions above. Check the line of sight port especially carefully.

- The heater on the ^{3}He pot may not be properly disconnected from its power supply (ITC). Check that the heater switching card on the back panel of the ITC is working correctly (ie. no volts across heater pins with 200 Ω across them).

- There may be a touch from the ^{3}He pot, the sample, or the wiring to the IVC; See if the ^{3}He pot temperature is affected by small changes in the VTI temperature. If all the other possibilities have been discounted, warm up the system and check the alignment.

- The excitation current for the high temperature sensor may introduce a small heat load onto the ^3He pot and warm it slightly. If a RhFe resistor is used it is best to unplug it from the ITC when operating the insert at the lowest temperatures.

4.5.3 Poor condensation

- Check the temperature of the SORB and 1K plate during condensation. The SORB should be set to the temperature recommended in the test results section of the manual. If the 1K plate is warmer than that appearing in the test results then there will be less 3He condensed and the hold time will be lower than that measured during system test at the factory. If the 1K plate is too warm, then try adjusting the flow of gas through the VTI to see if lower temperatures can be achieved there.

- Loss of ^3He, check pressure of ^3He at room temperature using a small volume gauge, it should be ~2bar.

If this is suspected, it is worth making some checks on the thermometry:

Try and check the base temperature with an independent thermometer - it is possible that the resistance thermometer has been damaged or its calibration has changed after a violent thermal shock.

- If the thermometry is correct, then it is possible that the SORB is contaminated, either contact Oxford Instruments for advice, or remove the ^3He from the insert and clean it using a cold trap, then pump the sorb using a diffusion pump while warming it to 50°C.

4.5.4 Poor pumping speed

4.5.4.1 Consistent leaks on the IVC cone seal

Check for scratches and polish them away carefully. Deep scratches in the male cone could be filled with low melting point solder (for example Wood's metal), and then polished. Check that you are using enough grease to seal the cone properly. A good fillet of grease should be visible after the seal has been made. It is helpful to rub the grease thoroughly into the metal surface.

4.6 Test results and system specific information

The test results for your system are given in a separate booklet (called "Heliox insert data") which includes all the information specific to your insert. In particular it gives details of:

- Experimental wiring
- Services fitted in the spare port
- Equipment supplied with your system and commissioning requirements

Operator's Handbook

Heliox Heater Controller

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Contents

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.

1 Introduction

The Heliox Heater Controller is an accessory for the ITC503 temperature controller to allow automatic operation of a Heliox System over a wide temperature range, without operator intervention.

Heliox includes two heaters, which are used in two different temperature regions. When operating below ~ 1.4K, a heater on the sorb is used to control its temperature and hence its pumping rate. This in turn controls the temperature of the ^{3}He pot. Above 1.4K a heater on the ^{3}He pot is used to warm the pot directly. Cooling of the pot then relies upon gaseous conduction through the ^{3}He gas to the 1K ^{4}He stage.

The simplest method of operation over both these regions uses a Heater Control Board (CQB1800) to switch the heater output of the ITC temperature controller to one or other heater as required. However this method has problems when operating in the high temperature region for extended periods. When no heater power is applied to the sorb heater, even with no cooling applied, its temperature will slowly fall to around 4.2K. This will effectively pump away all the exchange gas, which is required to cool the ^{3}He pot. Thus although it will be possible to heat up the pot, it will only cool very slowly.

The Heliox Heater Controller allows heat to be applied to the sorb heater even when controlling using the ^{3}He pot heater.

2 Installation

The Heliox Heater Controller is a free standing box which should be placed at the rear of the ITC503. In use the box may become warm, and so should be placed in a ventilated position.

Four flying leads on the heater controller should be plugged into the three CHANNEL inputs and the AUXILIARY socket on the rear of the ITC. Four corresponding sockets are fitted to the rear of the heater controller and the leads from the Heliox itself, should be plugged into these. For details of the pinouts of all these connectors, refer to the standard ITC503 manual.

2.1 Warning

The Heliox Heater Controller is intended quite specifically for use with a Heliox System having a Sorb Heater resistance of 30 Ohms or greater and an ^{3}He Pot Heater resistance of 100 Ohms or greater. The ITC503 should have been configured for a heater voltage limit of 30V or less (normally 25V for a Heliox). **Attempting to operate the unit with lower heater resistances than these or with a heater voltage of >30V may cause damage.**

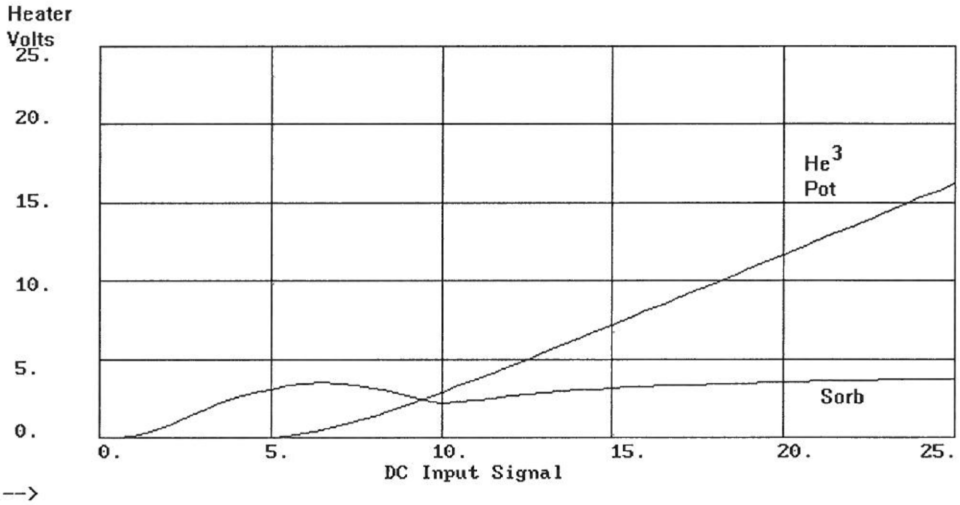
3 Operation

A Heliox is normally configured with the Sorb Temperature Sensor and Sorb Heater on Channel 1 and the high temperature ^3He pot Sensor and Heater on Channel 3. Channel 2 is normally used to monitor the low temperature ^3He pot sensor or the 1K pot temperature sensor and there is no heater associated with this channel.

There are no user controls on the Heater Control Unit. So it is only necessary to operate the Heliox via the ITC503 in the normal way.

When condensing the ^3He into the ^3He pot, ITC503 will be set to CONTROL on CHANNEL 1. This controls the sorb temperature, using the sorb sensor. In this mode of operation the Heater Control Unit will route all the ITC503 heater power to the sorb heater and there will be no heat applied to the ^3He pot.

For high temperature operation of the Heliox, the ITC503 will be set to CONTROL on CHANNEL 3, using the high temperature sensor on the ^3He pot. In this mode of operation the ITC503 heater power is distributed between the Sorb and the ^3He pot heaters. The graph below shows the typical behaviour. At low heater voltages, all the power is delivered to the sorb heater and no power is applied directly to the ^3He pot. This region may be used for control below 1.4K if allowed by the calibration range of the high temperature sensor (this is not usually the case). Control will typically require 2 to 3.5 volts applied to the sorb heater to maintain the sorb in the 10K to 30K region. If it is found that more voltage than this is being required to maintain the sorb temperature, the needle valve controlling the sorb cooling has been opened too much and liquid helium is being wasted.



When operation above 1.4K is required (which is the usual case when using the high temperature ^3He pot sensor) it becomes necessary to apply heat to the ^3He pot. To achieve this the ITC503 output voltage rises above 5 V and heat starts to be delivered to the pot heater. At the same time the voltage applied to the sorb heater falls, allowing this to cool slightly. However it is not allowed to fall to zero, so ensuring that the sorb remains warm enough not to pump away all the ^3He exchange gas. Thus cooling is still available, to maintain stable control.

In the change over region between the two modes of operation there is a smooth transition in output voltage between the two heaters, so allowing smooth control throughout this region.

As the required temperature is further increased the power to the pot continues to increase whilst the sorb power remains approximately constant. At high temperatures the Heliox will be operated with no cooling on the sorb. There is the risk that even the small residual sorb voltage would be sufficient to overheat the sorb and its wiring under these conditions. To avoid this, an additional control circuit is built into the unit. It senses the approximate sorb temperature directly, by monitoring the sensor current. (ITC503operates conductance sensors at fixed voltage, variable current). If the sorb temperature rises above 70K and the ITC is delivering more than 2 or 3 volts to the ^{3}He pot heater, all power is removed from the sorb heater. (Not shown on the graph).

With the control mechanism as described, steady state control is possible throughout the Heliox operating range. There is only one condition where intervention may be required. If a sudden large temperature decrease is required in the >1.4K region, the initial response of the ITC will be to reduce the heater voltage to zero. Depending upon the thermal masses attached to the ^{3}He pot, it is possible for the sorb temperature to fall to 4.2K (so removing all the exchange gas) before the pot temperature has fallen. Hence there will be no ^{3}He in the pot, to allow normal operation. To ensure this does not happen, whenever a large temperature fall is required, the Heliox should be operated initially in condense mode until the pot temperature is close to the required temperature (if this is above 1.4K) or has reached the normal "Condense Complete" conditions if the required temperature is below 1.4K. This will happen completely automatically if the Heliox is being driven from a computer using a version of the Oxford Instruments B-T environment software designed for use with the Heliox Heater Controller.

4 Servicing

4.1 Circuit Description

The ITC503 heater voltage enters on pins 6 and 7 of any of PL1 to PL3. At low heater outputs, the heater voltage passes via Q3 to the sorb heater (typically 60 Ohms) connected to SK1. R3 provides base current for Q3 and the result is that about 1.5 volts is dropped across Q3. Q2 does not conduct, so the voltage across R1 is less than the forward voltage of D4. Hence no current is delivered to the ^{3}He pot heater.

As the ITC output increases, a voltage is reached at which Q2 starts to conduct. Current then flows via D4 to the ^{3}He pot heater. The voltage dropped across Q2 remains constant thereafter (set by RV3), so the pot heater voltage increases linearly with ITC output voltage.

As the output further increases. Q1 starts to conduct, fed via R2. This pulls the base of Q3 negative, so reducing the sorb heater voltage. The value of RV1 determines the slope with which this voltage falls as the ^{3}He pot voltage rises. Eventually the voltage developed across RV2 is sufficient to cause Q4 to conduct. This then holds a constant voltage at the emitter of Q1. After this point, Q1 remains in saturation and the voltage at the base of Q3 and thence the sorb heater voltage remains constant.

During the ^{3}He condense operation, ITC503 is set to control on Channel 1. Under these conditions the cathode of D2 is at a logic high. Current from the 5V rail is able to flow via R5, D3 and turns on the opto isolator U1. This in turn clamps the upper end of RV3 close to ground, preventing a ^{3}He pot heater output and allowing the sorb heater to receive the full output voltage. When the ITC503 is set to control on Channel 2 (the low temperature ^{3}He pot sensor) the sorb heater also receives the full output voltage. However, in high temperature operation, control will be on channel 3, so that the current from R5 will be diverted through D2 and U1 will remain off.

The sensor current for the 100 Ohm Allen Bradley sensor on the Sorb, passes through the opto isolator U2. This allows a secondary current to flow through RV4 which is a function of the sorb temperature. (The extra voltage drop across the primary of U2 has no effect on the normal ITC503 sorb temperature measurement). When the voltage across RV4 is sufficient to turn on Q5, the base of Q3 is brought close to ground and the sorb heater voltage is reduced to zero. This forms the high temperature protection for the sorb. R6, D5 provide a stabilised voltage for this circuit.

4.2 Adjustments

The 4 internal preset potentiometers are set during manufacture and system test for best results with a Heliox system. They should not normally require re-adjustment.

The normal adjustment conditions are described below, but these may be varied to suit individual systems.

Adjustments are made by applying a measured input voltage from an ITC and adjusting to obtain a specified sorb or ^{3}He pot output voltage. Note that the ITC voltage should be measured. The nominal output voltage set on the ITC will not be sufficiently accurate. The adjustments should be carried out in the order given. On a completely new unit all four potentiometers should be set fully clockwise. When making small changes to an existing unit, the previous settings are an acceptable starting point.

4.2.1 RV3

Set the ITC output to 6.0V (measured across C1). Adjust RV3 to obtain a ^{3}He pot heater voltage of 0.33V (+/-0.03V) measured from the cathode of D4 to the -ve side of C1.

4.2.2 RV1

Set the ITC output to 8.0V (measured across C1). Adjust RV1 to obtain a sorb heater voltage of 2V (+/- 0.4V) measured from the emitter of Q3 to the -ve side of C1.

4.2.3 RV2

Set the ITC output to 20V. Adjust RV2 to obtain a sorb heater voltage of 3.5V (+/-0.7V). If large changes are made to RV2. It may be necessary to repeat the adjustment of RV1, till both are correct.

4.2.4 RV4

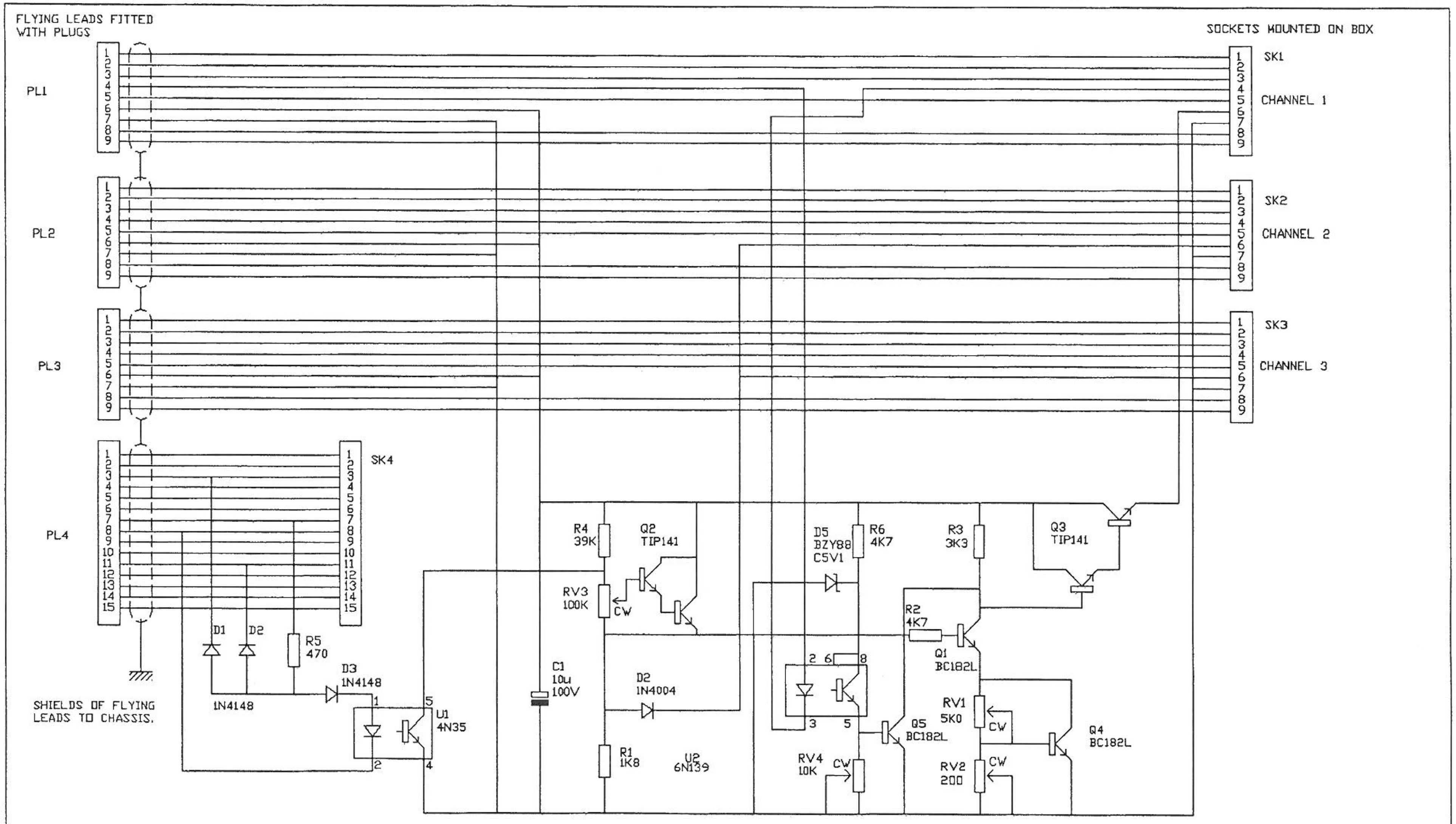
With the ITC output set to 12V and the CONTROL CHANNEL set to CHANNEL 1, monitor the sorb output voltage. (It should be around 11V). Substitute a resistance box for the sorb temperature sensor and adjust this for a displayed sorb temperature of 70K +/- 5K.

Adjust RV4 until the sorb voltage falls to around 5V. The fall will occur very sharply as RV4 is varied and it is not critical to set to voltage accurately to 5V. It is sufficient to set RV4 to the point where the falling edge occurs.

4.3 Circuit Diagrams

The following circuit diagram is included in this section.

Drawing No.	No. of pages	Description
	(1 sheet)	HELIOX HEATER CONTROLLER



ISSUE 3	AFS 12/1/96	D5, R6 ADDED. R1 VALUE CHANGE.
ISSUE 2	AFS 9/1/96	U1 ADDED
ISSUE 1	AFS 27/11/95	BASIS FOR MODELLING

HELIOX HEATER CONTROLLER

OXFORD

DRAWING NUMBER

A4/