Chase
Research
Cryogenics

World leaders in
sub-Kelvin cryogenics

TWO-STAGE SUB-KELVIN ^3He SORPTION COOLER

TYPE GL7 (Helium 7)

GENERIC INSTALLATION AND OPERATING INSTRUCTIONS

Photo shows a typical CRC GL7 (Helium 7) sorption cooler

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Contents

THIS GENERIC OPERATING MANUAL describes how to install and operate a CRC GL7 sorption cooler. It is accompanied by an Excel file that contains the validation test data and the calibration files that are **specific** to the sorption cooler unit that you have purchased.

Both this manual and the Excel data file are important parts of the technical documentation for this product. You are advised to make a note below of the location of the Excel file specific to your sorption cooler unit. CRC can, on request, provide a copy of the Excel file for your unit – just let us know the serial number engraved around the mainplate.

This revision of the manual was created in May 2022.

1. GENERAL HANDLING

WARNING!

CRC SORPTION COOLERS CONTAIN HELIUM GAS AT HIGH PRESSURE.

Do not crush, twist or bend the unit. Avoid applying mechanical stresses. Do not heat the unit above room temperature. Keep in a sealed cryostat, or in the shipping box and brace in which it came.

Do not hold or lift the unit by the cold heads.

Do not tamper with the copper capillary fill tubes.

Avoid the use of acid fluxes when soldering near the sorption cooler. Chloride based fluxes will corrode stainless steel and could damage your sorption cooler.

Always allow the unit to warm to room temperature before allowing air into your cryostat. Opening the air valve of a cold cryostat is likely to damage the unit

After unpacking the sorption cooler according to the instructions supplied, the sorption cooler should be immediately transferred into the host cryostat. The shipping brace doubles as a stand for the sorption cooler, though when used as a stand, the screws through the aluminium plate into the cold heads should NOT be in place. When picking the sorption cooler up, it should be held by the main plate.

2. SAFETY OF CHASE RESEARCH CRYOGENICS PRODUCTS

2.1. Pressure Equipment Directive 2014/68/EU.

This CRC sorption cooler unit is manufactured in accordance with Sound Engineering Practice. The volume and gas pressure within the sorption cooler are such that the equipment falls below the lower classification limit in Annex II of the Pressure Equipment Directive. Hence the requirements for Conformity Assessment do not apply and no Declaration of Conformity can be made, or CE marking applied.

The sorption cooler is covered by Article 4 Paragraph 3 of the Pressure Equipment Directive, which states:
"Pressure equipment and assemblies below or equal to the limits set out in points (a), (b) and (c) of paragraph 1 and in paragraph 2 respectively shall be designed and manufactured in accordance with the sound engineering practice of a Member State in order to ensure safe use. Pressure equipment and assemblies shall be accompanied by adequate instructions for use. Without prejudice to other applicable Union harmonisation legislation providing for its affixing, such equipment or assemblies shall not bear the CE marking referred to in Article 18."

2.2. Pressure Equipment (Safety) Regulations 2016.

The pressurized modules making up this sorption cooler unit have internal volumes much lower than 1 litre, and pressure x volume much lower than 200 bar-litres, hence the sorption cooler is exempt from the Essential Safety Requirements set out in Schedule 2 of the PESR Regulations 2016. This means that the sorption cooler does not require a written scheme of examination. The sorption cooler complies in all respects with the requirements of Regulation 8 of PESR 2016. The *owner* has duties under the PESR regulations to ensure that a) the safe operating limits are not exceeded; b) the unit is operated in accordance with these instructions; c) the unit is returned to Chase Research Cryogenics Ltd in the event that any maintenance is required. The sorption cooler contains no user-serviceable parts.

2.3. Safe Operation

The safe operating temperature range of this sorption cooler is 0 to 320 K.

2.4. Risk Assessment

CRC sorption coolers contain Helium gas under pressure. The stored energy of the system is less than 50 bar litres. All system components are integrity tested during manufacture; the slightest leak will make the sorption cooler lose its stored gas and cease to function. A unit that has leaked presents no risks whatever to the user; the following risk assessment applies therefore only to functional units.

Hazards and consequences

Accidental damage to the sorption cooler unit could result in the sudden release of pressurised gases, causing mechanical failure of the unit and potential injury (or damage to surrounding instruments) from ejected debris. Possible events leading to failure are: overheating of the unit, for example in a fire; dropping or crushing of the unit; twisting or bending of the gas tubes. Mechanical damage to the unit is most likely to occur during assembly of the instrument of which the sorption cooler forms part.

Risks without controls in place

It is extremely unlikely that the above events will lead to danger. Chase Research Cryogenics Ltd has produced several hundred sorption cooler units of various designs, which are in use for a range of applications worldwide. To date there has never been a sudden failure of a sorption cooler unit – indicating that with normal use (including inevitable handling mishaps) the units have an excellent safety record. User experience to date shows that accidental mechanical damage to sorption cooler units is likely to result in slow leaks, not sudden failures.

Controls in place

The controls that are in place to eliminate (as far as reasonably practicable) the risks arising from mechanical damage to a sorption cooler unit are:

- This written instruction manual, containing warnings about the potential risks arising from damage to the unit and alerting the user to more risky operations;
- Instructions that the unit should not be used if it has been subjected to overheating, dropping, crushing, bending or twisting;
- A warning in the unpacking instructions that this operating manual should be read prior to using the unit.
- A shipping brace that can be used to store the unit safely when not in use.

The applications for which sorption cooler units are intended make it impossible to place warning labels on the unit itself. However if the sorption cooler is incorporated into another instrument, that instrument should carry a warning label to alert the user that the sorption cooler contains no user-serviceable parts and should not be disassembled.

Risks with controls in place

Providing users read and follow this instruction manual the risks are negligible.

2.5. Safe Transportation

The unit must be correctly re-installed in its shipping brace before transportation either by road or air. Follow the unpacking instructions provided in reverse order and contact CRC for advice if needed. The cooler+brace should be securely packed into the cooler's rigid shipping box, which contains polystyrene or similar cushioning material to firmly hold the unit and prevent any movement within the box. Add extra pieces of packing foam to ensure that the heavy pumps cannot move in transit. Place the rigid shipping box into a much larger outer carton surrounded by at least 5-10 cm soft fill on all sides, this should ensure that if dropped the impact shock will be absorbed by the overpack, and not transmitted to the cooler itself.

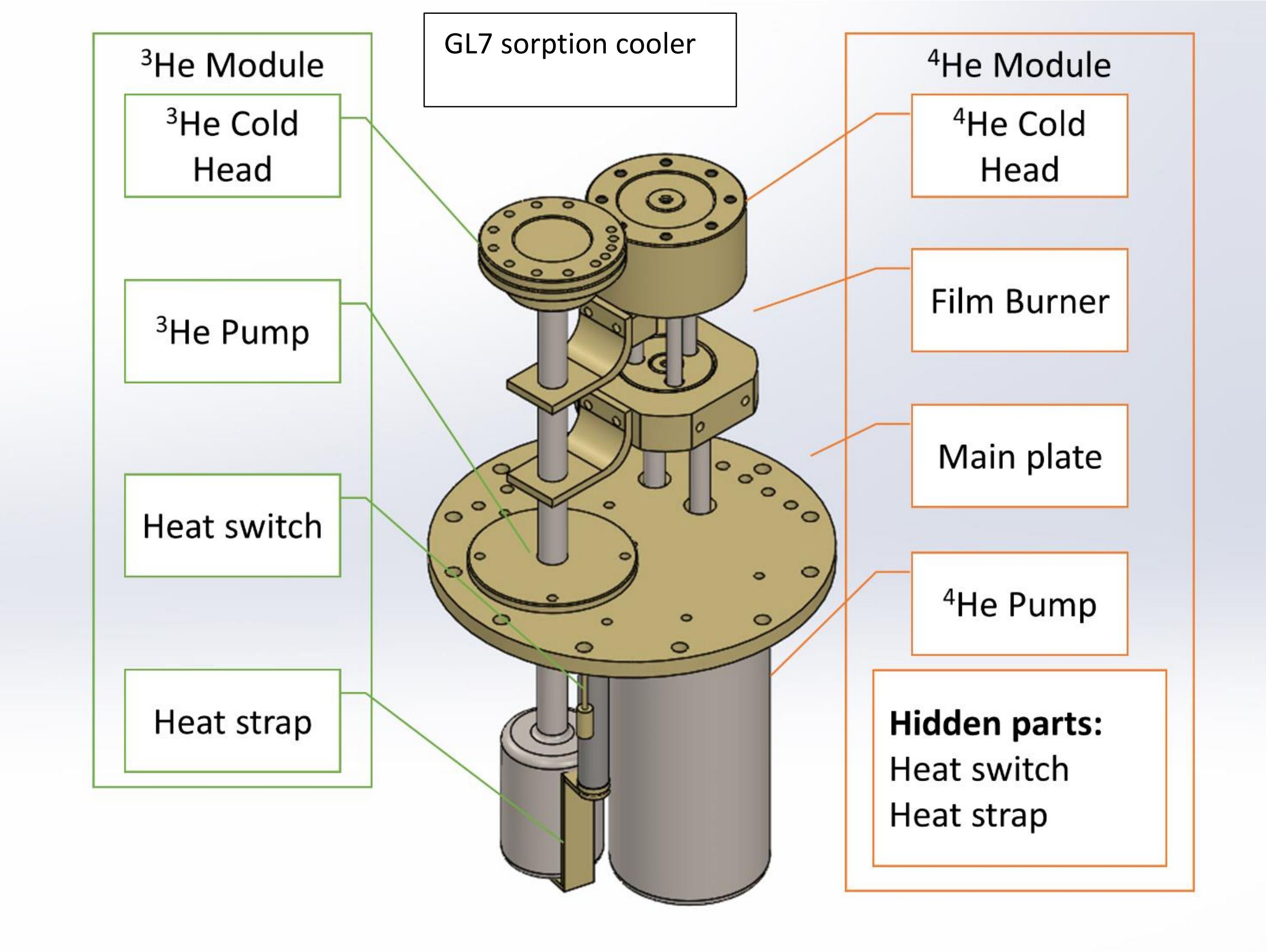
For transportation purposes CRC sorption coolers are classified under UN 2857, Class 6A (6=Other articles containing gas under pressure; A=asphyxiant gas). Special Provision 119 applies: *Refrigerating machines include machines or other appliances which have been designed for the specific purpose of keeping food or other items at a low temperature in an internal compartment, and air conditioning units. Refrigerating machines and refrigerating machine components are not subject to the provisions of ADR if they contain less than 12 kg of gas in Class 2, group A or O according to 2.2.2.1.3, or if they contain less than 12 litres ammonia solution (UN No. 2672).*

This special provision means that the cooler does not need to be either labelled or shipped as hazardous or dangerous goods, whether transported by road or by air, provided that it is appropriately packed.

Ensure that the outer carton is clearly labelled as fragile with 'This Way Up' labels and add Shock Indicators if you are handing the consignment to third-party couriers, as these should deter rough handling.

3. A BRIEF DESCRIPTION OF THE SORPTION COOLER UNIT

The sorption cooler shown in the figure below is a standard model. In use, the sorption cooler is inverted, i.e. the heads will be at the bottom. The main plate needs to be thermally sunk to the cold head of a cryocooler at 4K or below, see section 4.1 for more information. The ^{3}He head, ^{4}He head and the film burner can all be used to extract heat from the user's experiment, see section 5 for more information. The pumps and heat switches can reach up to 50K during operation, these need to be radiation shielded from the heads, for more information see section 5.1. All electrical connections are brought out to an MDM connector mounted onto the main plate. Pin-outs are listed at the end of this manual.



The following short names for the various parts of the sorption cooler are used throughout this user manual:

Short name used in this manual	**Refers to the sorption cooler part**
3-head	^{3}He cold head
4-head	^{4}He cold head
FB	Film burner
3-Pump	^{3}He pump
4-Pump	^{4}He pump
3-Switch	Heat Switch for the ^{3}He pump
4-Switch	Heat Switch for the ^{4}He pump

4. INSTALLATION

4.1. Mechanical

Before installing the unit in your cryostat, be sure to remove all pieces of foam packing material from around the pumps, as mentioned in the unpacking instructions.
There should be no need to touch the heat switches or heat straps during installation or normal operation of the sorption cooler. The heat switches can be easily damaged, and if bent or twisted are likely to fail.

This sorption cooler is designed to work equally well in either 'wet' cryostat using liquid ^{4}He to cool the mainplate, or in a 'dry' cryostat with the mainplate thermally sunk to a mechanical cryocooler at 4K, such as a GM or pulse tube sorption cooler. The 4K stage of the cryocooler should be made from gold plated copper to ensure excellent thermal contact between the sorption cooler and the cryocooler. To attach the sorption cooler to the 4K stage of the cryocooler there are twelve 4.1mm diameter (M5 clearance) holes symmetrically distributed upon a 115 mm pitch circle around the periphery of the circular main plate. In addition to these, there is also a row of 5 x M4 clearance holes at ½" (12.7mm) centres, close to one edge of the main plate. (Note: UNC #6 holes are substituted if requested by the customer). A .step CAD file of your sorption cooler can be provided on request.

Because the cooling down of the heads depends upon gas convection, and on liquid helium collecting in the heads fed by gravity, the sorption cooler *mus*t be kept close to vertical with the heads downwards.

Ensure spring washers are under every bolt head, these will take out differential thermal contraction that might otherwise cause loosening of the bolts, and thus compromise thermal contact.

4.2. Electrical

All electrical connections are on a 25-pin MDM-SSP connector mounted onto the main plate. Pin-outs are listed at the end of this instruction manual. The table below summarises the temperature sensors installed on the unit.

ITEM	Calibration	Options
3-head RuO_2	Generic Lakeshore Cryotronics or Scientific Instruments.	Individually calibrated sensors available on request
4-head RuO_2	Generic Lakeshore Cryotronics or Scientific Instruments.	Individually calibrated sensors available on request
Film burner diode	Specific calibration supplied in individual data file	No diode supplied
Pump diodes	Generic – supplied by CRC Ltd	
Switch diodes	Generic – supplied by CRC Ltd	

Voltage / current requirements for driving the heaters and thermometers are summarised in the table below.

ITEM	NUMBER	IMPEDANCE/ JUNCTION VOLTAGE	VOLTAGE/ CURRENT
3-pump heater	1	300Ω approx.	20 to 25V
4-pump heater	1	300Ω approx.	20 to 25V
Heat switch heaters	2	10kΩ	4 to 5V
Diode thermometers	5	0.5 to 1.8V	10μA DC
4-head RuO_2 thermometer	1	1kΩ to 3kΩ	1μA max.
3-head RuO_2 thermometer	1	1kΩ to 7kΩ	100nA max.

Generic (i.e. standard calibration) RuO_2 sensors from Lakeshore Cryotronics or Scientific Instruments Inc are the default option on the heads of all CRC sorption coolers. Individually calibrated 'CERNOX' or RuO_2 sensors are only fitted (at additional cost) at the customer's express requirement. The thermometer on the 3-head is operated as a 4-wire device and should be excited with an AC current no greater than 100nA, corresponding to a voltage of around 2mV at base temperature. The thermometer on the 4-head is operated as a 2-wire device and should ideally be driven by an AC current no greater than 1μA. A reasonable temperature estimate can be gained by driving this sensor with 10μA DC, though this is likely to cause some self-heating and can also be vulnerable to thermo-electric DC offsets, particularly at higher temperatures.

Calibration data for all thermometer sensors are in the Excel data file that accompanies each unit. Generic diode calibration curves for the pump diodes and heat switch diodes, and a calibration curve specific to the film burner diode, are supplied as standard. The diode thermometers require excitation with currents of 10μA DC.

The heat switch heater typically requires about 4 to 5 V to keep the switch in the 'ON' state with the absorber pod at greater than ~20K, and it will cool to the off state (T < 10 K) in ten to fifteen minutes.

The pump heater impedances are typically 300Ω. During the cooling cycle it is necessary to warm the 4-pump to around 50 to 60K and the 3-pump to 45 to 50K. A heater current of up to around 100 to 130mA for the 4-pump, and around 50 to 60mA for the 3-pump, will heat the pumps rapidly; lower heater currents will result in slower heating. Stabilisation of the pump temperatures at around 50K will typically require heater currents of around 12 to 15mA. Try to ensure that the lead-in wiring to these heaters is not unduly dissipative.

5. ATTACHING YOUR EXPERIMENT TO THE SORPTION COOLER.

This model of sorption cooler provides three points at which heat may be extracted from a user's experiment mounted on a separate cold table. They are the 3-head, the 4-head and the film burner. To achieve optimum performance, only a very small load should be applied directly to the 3-head. The main source of cooling power is the 4-head, which can sustain a thermal load of at least 250μW at a temperature of less than 1K. The film burner may also be used to sink some load at around 2K.

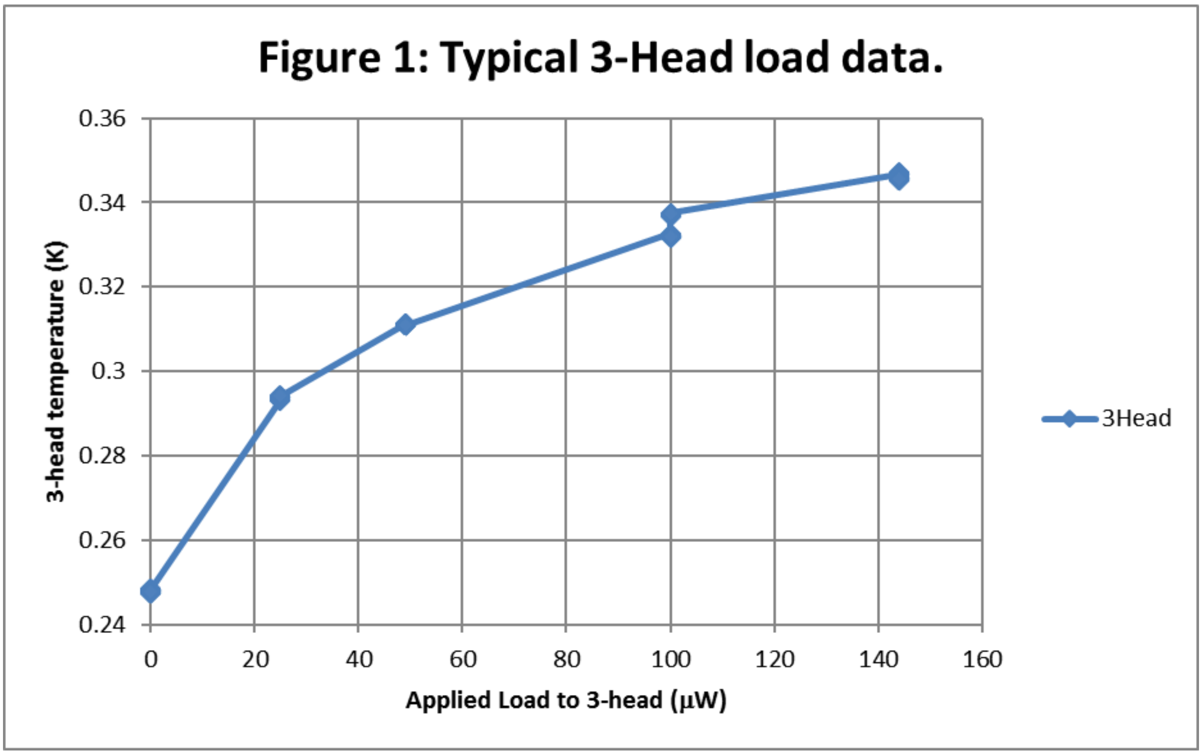
The 3-head has 9 tapped M3 holes on a 40mm pitch circle. The 4-head has 8 holes tapped M3 on a 40mm pitch circle and a further axial hole tapped M4. The film burner has 6 M3 tapped holes on the main body, in pairs on each of the three free sides 20mm apart. (Note: UNC #4 threads will be substituted if requested by the customer). A step file can be provided on request.

While fixing experimental equipment to the cold heads, extreme care should be taken not to twist or bend the gas pipes. Always support the cold heads against the applied torque.

The size of a GL7-type sorption cooler determines its run time and temperature at a given heat loading. The sorption cooler you have purchased will have been built to your specified customer requirements and tested to verify that it meets its specification. Details of the specification and the test results are given in the Excel file accompanying your unit. Typically, the run time is limited by the 4-head, which will have been built to support the anticipated load requirements in the user specification. Under no load the 3-head will typically run at about 260mK, the 4-head at about 865mK, and the film burner at about 1 to 1.6K. When loads are applied, the heads and film burner naturally run warmer, see Figure 1. Load data for your specific sorption cooler are included in its accompanying Excel test data file.

5.1. Radiation shielding

The cold heads, and any cold table/experimental equipment/detector assembly you attach, must be properly radiation shielded at around 4K in order to achieve sub-Kelvin operation. Any ancillary support structure (cold table) and experimental wiring looms must be thermally sunk to the 4-head to improve the operating temperature. Temperatures below around 300mK are only achievable if the total thermal load on the 3-head is kept as low as possible. The 4-head and film burner are designed to buffer the parasitic loads due to wiring and mechanical support structures. No other attachments to the sorption cooler unit are necessary for achieve satisfactory operation. If your sorption cooler performance is not meeting the specification you expect, this is likely to be due to a radiation load on the sorption cooler. Check your radiation shielding and consider adding extra multi-layer insulation around your radiation shields, or around the pump.



6. OPERATION: QUICK-START

6.1. Summary of the operating steps

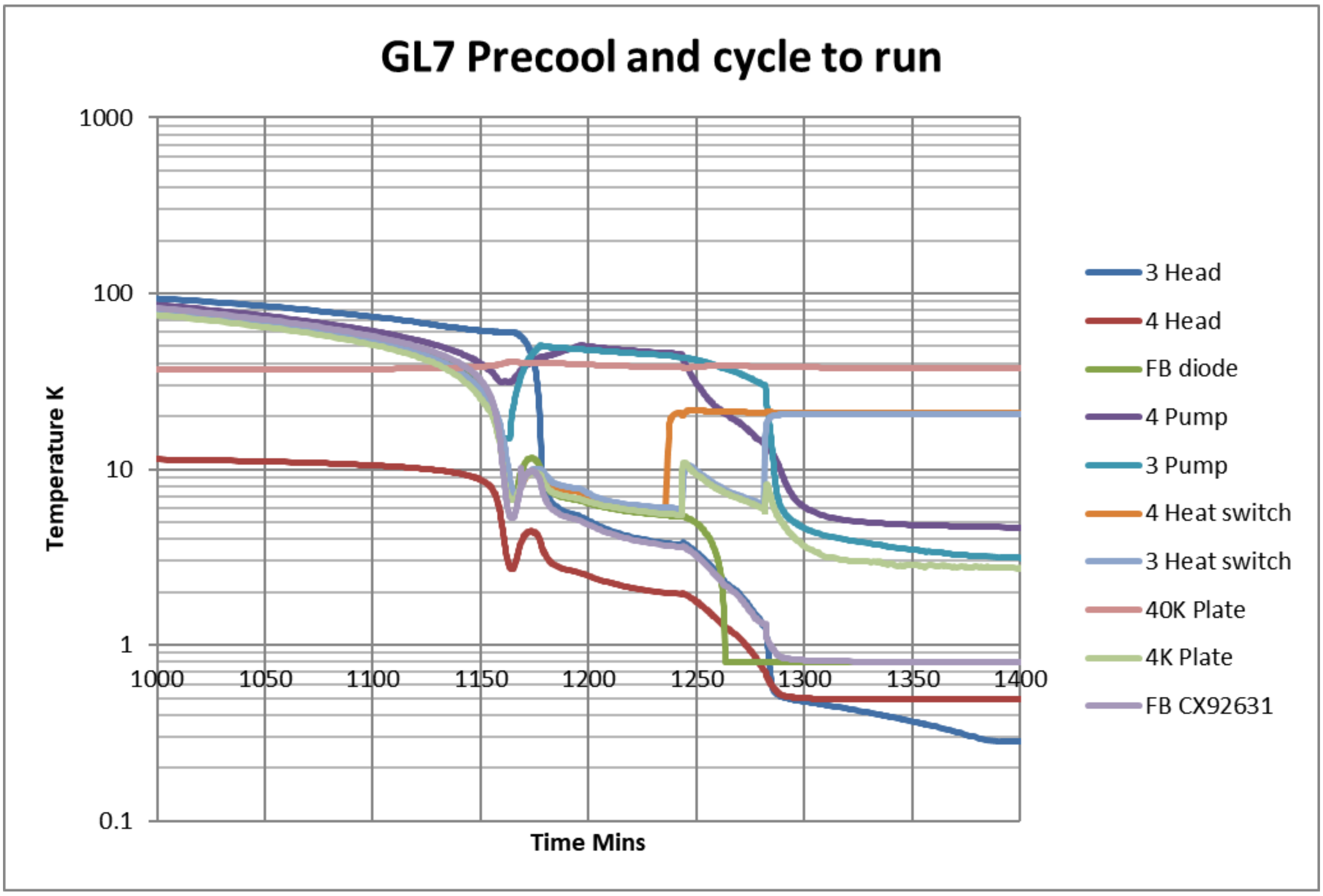
The basic operational sequence is as follow.

- Pre-cool from room temperature to ~4K
- During pre-cooling, when both heat switches turn OFF (they are at less than 10K), heat both pumps to around 45-55K and keep them at that temperature until the heads cool to ~4K and their temperature stabilises.
- Turn OFF the 4-pump heat and turn ON the 4-switch.
- When the heads have cooled to less than 2K, turn OFF the 3-pump heater and turn ON the 3-switch.

A detailed flow diagram showing all the steps for running the sorption cooler is included at the end of this manual. The temperatures suggested are only approximate and may need to be adjusted to achieve the best performance for your specific sorption cooler and experiment. If you are using a mechanical cryocooler with a low cooling power, try the lower end of the suggested temperature range. If you are using a more powerful mechanical cryocooler, or precooling with liquid helium, you will get the best performance at the top end of the suggested temperature range. The operational sequence for a typical GL7 sorption cooler is illustrated below.

6.2. Pre-cool and cycle to run

An illustration of a typical pre-cool and first cycle with a low-power GM cryocooler is shown below. Cooling times will be shorter with a higher-power GM.



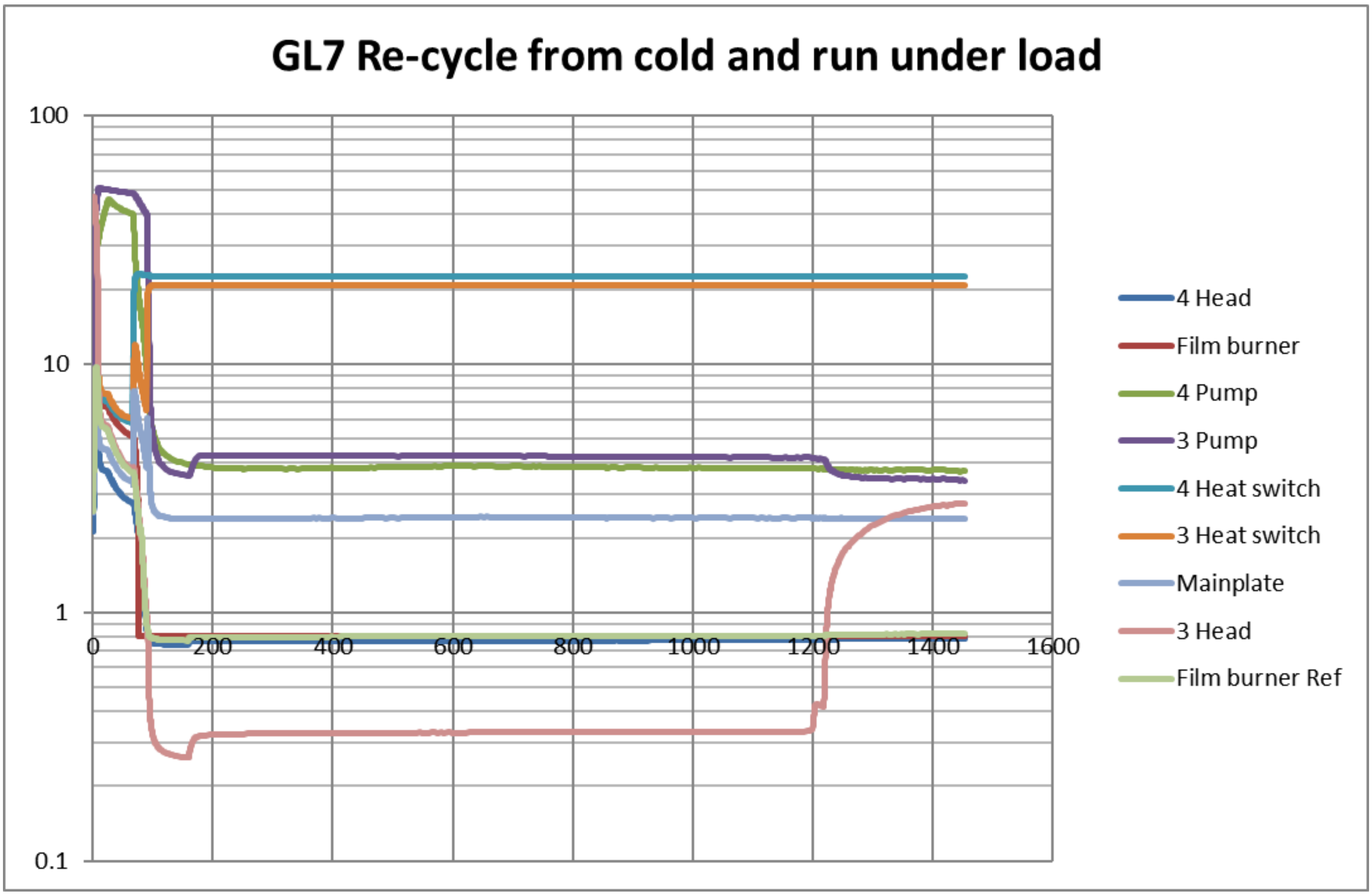
Here you can see that after ~1160 minutes the 4-head temperature is less than 4K, the heat switches are off (<10K). To get the GL7 running the pumps are both warmed to more than 45K by applying a voltage of 20V. The pump heaters are turned down or off while the 4-head temperatures drop to ~2K. The 4-heat switch is turned on with a voltage of 5V and the head temperatures fall rapidly below 1K. Turning on the 3-switch then starts the final cooldown of the 3-module to ~300mK.

Note in this figure that the film burner diode readings bottom out during the run because the generic diode calibration has been used. Diode readings at these temperatures are only indicative. In some cases we are able to provide a specific calibration for the film burner diode fitted to your sorption cooler, which will improve the accuracy of the FB diode readings below 4K.

6.3. Recycle from cold and run

The run time of a GL7 sorption cooler will depend on the size of the unit (i.e. how much gas it contains), on parasitic loads, and on loads applied by your cryostat and experiment. When the unit expires, it can be recycled from cold and run again. A typical sequence with a low-power GM cryocooler is illustrated below.

At the beginning of this sequence the 4-head has expired and the heat switch has been turned off. As before the pumps are warmed to nearly ~45K and the 3-pump is kept warm while the 4-head re-cools and stabilises. The 4-switch is turned on to cool the 3-head. The 3-switch is then turned on and the GL7 once again begins to run. The entire recycling sequence takes around 100 minutes. A faster recycle (warming the pump rapidly with a higher voltage) can be carried out if a higher-power GM is used, as it has more capacity to quickly remove the heat input to cycle the pump.



7. HINTS AND TIPS FOR OPTIMISING THE PERFORMANCE OF YOUR SORPTION COOLER

7.1. Pre cool

Keep the pumps above 25K once the switches have turned OFF for the initial cooldown.

It is important to understand that for the initial cool down, the cold heads cool by gas convection, and the pumps cool by conduction via the heat switch while the heat switch is ON. The cold heads will cool rapidly while the pumps are warmer than ~25K, but once the pumps drop below this temperature the heads could take up to a few days to reach the final cooldown temperature. This is because when the gas is adsorbed into the pumps, the heads cannot cool by gas convection. The key to a rapid cooldown is: once the heat switches have turned OFF, reheat the pumps above ~25K and stabilise them at this temperature. You should then see the cold heads cool rapidly to around 4K.

To reduce the load on your cryocooler (or usage of liquid cryogens in a wet dewar), time the heating of both pumps so that they reach their target temperatures at the same time. This will also give you a faster cooldown.

7.2. Running the 4He module

Experiment with varying the pump temperature during the run to find the best performance for your set up.

Once the 4-head is ~4K, and the switches are off, the sorption cooler is ready to start running. The generic method is to heat the pumps to the suggested temperature in the table in section 6.1 and maintain them there whilst ensuring the 4-head cools to below the critical liquefaction point of ^{4}He (5.2K). However the colder the 4-head gets while the pumps are hot, the higher the liquefaction efficiency, and hence the longer the sorption cooler will run before it must be recycled. You should try variations of pump temperatures to find a procedure that provides the best performance for your set up. When operating from a low-powered mechanical cryocooler (e.g. 100mW @ 4K PT unit) you will probably achieve more efficient ^{4}He condensation by starting at the lower end of the suggested range of temperatures. This is because imposing smaller load on the mechanical cryocooler from the hot pumps will enable a faster and more efficient recycle and run.

7.3. Operating the heat switch on the 4He module

Turning the switch on slowly will put less load into the cryocooler.

Once the ^{4}He is liquified (when the 4-head and film burner temperatures have stopped falling) the 4-pump is allowed to cool by turning OFF the pump heater and turning ON the heat switch. The hot 4-pump will impose a large heat load onto the 4K plate, this can cause the temperature to rise temporarily. However, the rate of cooling the pump can be varied depending on the voltage applied to the switch. The switch will begin to turn ON at 14-17K and be fully on above ~20K. If the switch is turned on slowly, by applying a lower voltage at first and gradually increasing it, the heat from the pump is dissipated more slowly and so there is less temperature rise at the 4K plate. In addition, if there is a small pause between turning off the pump power and turning on the switch power, in this time the pump will cool slightly by the parasitic load down the pump tube. At this point the 4-head temperature and film burner temperature will fall rapidly.

It is particularly important to control the rate of cooldown in a GL7 because if the main plate temperature rises too high there is a danger that the 3-switch will turn ON. It is important to keep the 3-pump warm (and the 3-switch OFF) until the 4-head temperature has dropped below 2K. The lower you can get the 4-head temperature before cooling the 3-pump, the better.

7.4. Operating the ^{3}He module

Wait to turn on the 3-switch till you see certain signals.

Once the 4 switch is ON you can turn OFF the 3-pump power, but only turn on the 3-switch when the 4-head is below 2K to maximise liquefaction efficiency of the ^{3}He. If running from a low powered mechanical cryocooler it is better to wait longer to turn on the 3-switch. The film burner closely follows the cool down of the 4-head until 0.9K, where it will stay for a short while before rising in temperature again. You should wait for this signal to turn on the 3-switch.

While the 3-pump is cooling the 3-head will also cool rapidly. Final stabilisation at the operating temperature will take some time; how long will depend on the thermal loads applied by your experiment. The 3-head can take some while to stabilise, particularly with applied loads of less than 1µW or so. This is because the liquid ^{3}He has a high specific heat capacity compared to the rate at which gas evaporation (at very low vapour pressure) can extract latent heat. The lower the final operating temperature, the lower will be the corresponding saturated vapour pressure, and the rate at which gas evaporates.

7.5. Parasitic loads

Use the 4-Head and Film burner to buffer any parasitic loads.

In operation, the parasitic loading may be distributed between the 4-head and the film burner in order to optimise the 3-head temperature or the run time. The longest run times will be obtained when the loads on the 4-head and 3-head are kept below about 150µW and 20µW respectively.

8. STANDARD PIN-OUT ASSIGNMENTS.

GL7 pinouts to 25-pin micro-D SSP

Function	Red box for twisted pair.	TYPE	female 25 pin PIN #	Drive current or voltage
3He HEAD THERMOMETER V+			**1**	AC bridge or
3He HEAD THERMOMETER V-			**14**	low-current driver
3He HEAD THERMOMETER I+			**2**	100nA typical
3He HEAD THERMOMETER I-			**15**	
n/c			**3**	
4He HEAD THERMOMETER V+			4	AC bridge or
4He HEAD THERMOMETER V-			16	low-current driver
DIODE FILM BURNER I+			5	10 µA Constant current,
DIODEFILM BURNER I-			17	Read Junction Voltage.
DIODE 4He PUMP I+			6	10 µA Constant current,
DIODE 4He PUMP I-			18	Read Junction Voltage.
DIODE 3He PUMP I+			7	10 µA Constant current,
DIODE 3He PUMP I-			19	Read Junction Voltage.
DIODE 4He PUMP SWITCH I+			8	10 µA Constant current,
DIODE 4He PUMP SWITCH I-			20	Read Junction Voltage.
DIODE 3He PUMP SWITCH I+			9	10 µA Constant current,
DIODE 3He PUMP SWITCH I-			21	Read Junction Voltage.
HEATER 4He PUMP SWITCH I+			10	10kΩ heater element
HEATER 4He PUMP SWITCH I-			22	4-5V supply (approx)
HEATER 3He PUMP SWITCH I+			11	10kΩ heater element
HEATER 3He PUMP SWITCH I-			23	4-5V supply (approx)
HEATER 4He PUMP I+			12	300Ω heater element
HEATER 4He PUMP I-			24	0-30V supply (approx)
HEATER 3He PUMP I+			13	300Ω heater element
HEATER 3He PUMP I-			25	0-30V supply (approx)

Resistance thermometer

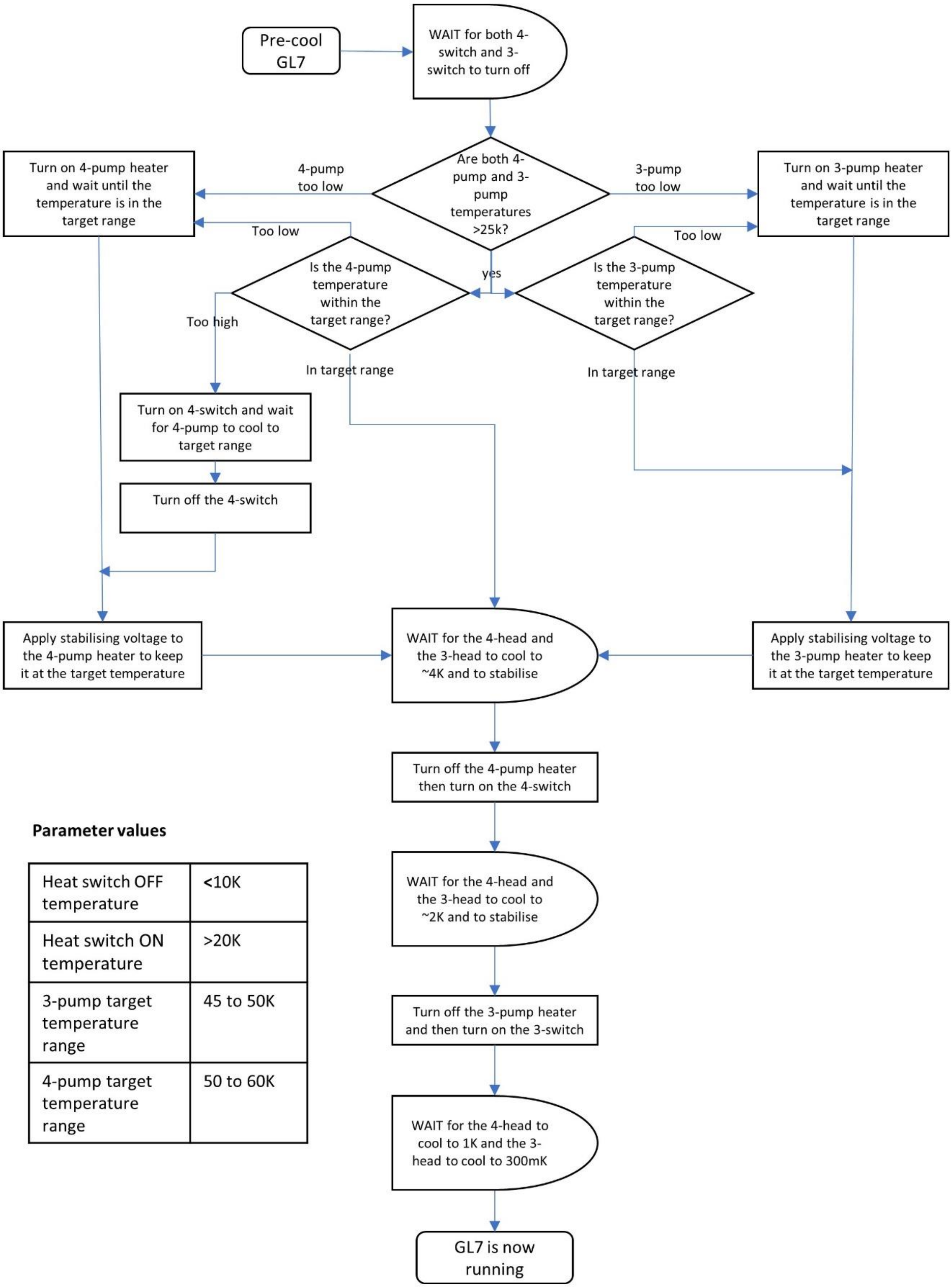
Diode thermometer

Low power heater (a few mW)

High power heater (up to about 2W)

9. FLOW CHART OF GL7 OPERATING PROCEDURE

Note that some operations occur in parallel.



Parameter values

Heat switch OFF temperature	<10K
Heat switch ON temperature	>20K
3-pump target temperature range	45 to 50K
4-pump target temperature range	50 to 60K