

The Genevac HT- 4 & HT- 4X Series II Evaporation Systems



User Manual

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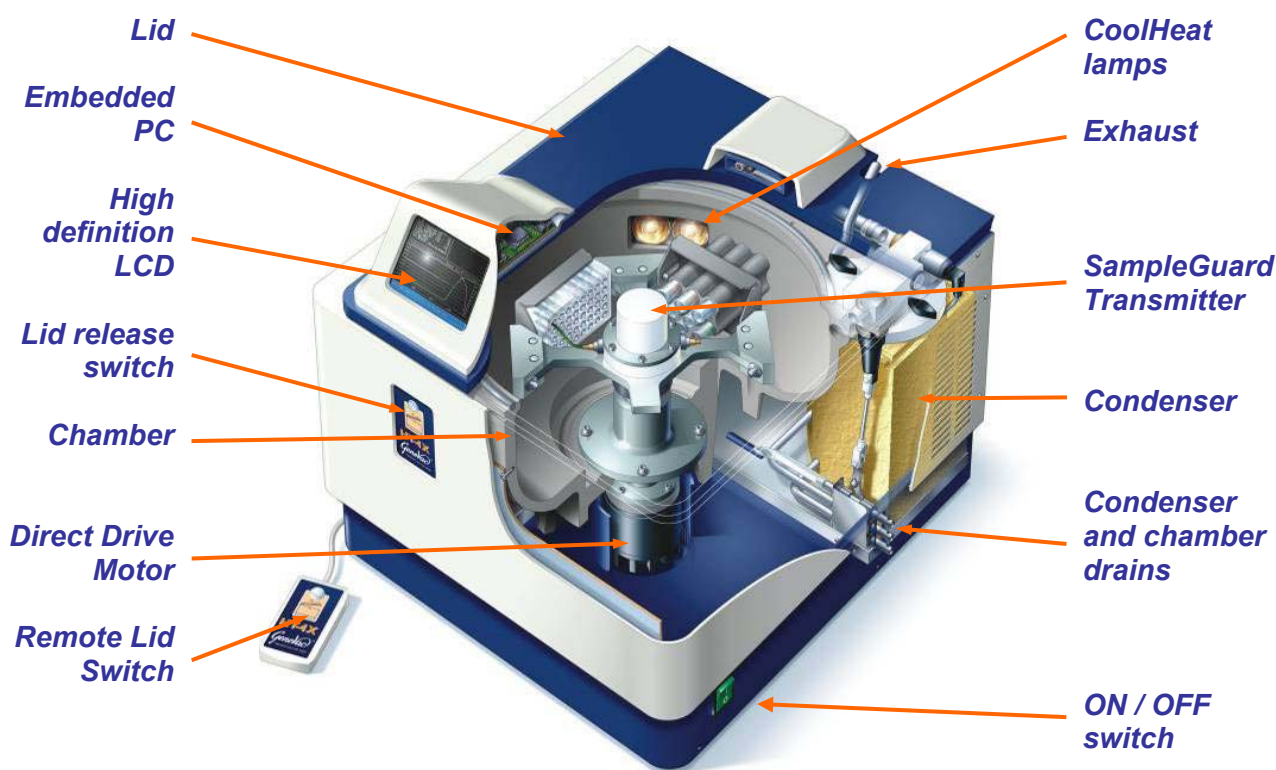
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Introduction

The Genevac Series II range of evaporation systems provide very high performance coupled with ease of use. The systems are simple to set up, easy to operate and very flexible. The **HT4-SII** and **HT4X-SII** evaporation systems are comprised of an evaporation chamber and rotor, combined with an in-built **cryopump-condenser** unit. Vacuum is provided by a **Scroll Pump** (although other types of vacuum pumps can be used).

Solid-state case heaters and CoolHeat radiant lamps heat the chamber and samples. The control of chamber, sample holder and sample temperature, vacuum ramping rate, chamber pressure, rotor speed and run time are all handled by an embedded PC.



The system offers the facility to auto program run parameters for any (recognised) solvent or mixture. Simply select the solvent(s) from a drop down menu, enter details for volume and sample holder type, select Auto Program and optimised run parameters appear highlighted in cyan on the display screen.

The status of the system is shown on an LCD screen. Programming input is via a keypad and simple to use up-down controls enable the run time, rotor and chamber temperatures to be set in an instant.

Lyophilisation (Freeze Drying) may be achieved in **LyoSpeed™** enabled systems fitted with an **Auto Defrost & Drain** condenser. Samples must be frozen before loading into the evaporator.

Automatic end of run shut down may be programmed manually with a simple time setting, or may be achieved by automatic end of run prediction using **Heat Flow** or **Sample Detection** methods.

The **Run Progress** screen has a fully featured graphical display, providing annotated plots for temperature and pressure. The control software enables the user to specify and store a library of up to 100 different evaporation profiles.



Evaporator Screen and Keypad

Run profiles can also be linked to provide multi stage runs. Up to 100 different profile steps can be linked and iterative loops can also be programmed. With a maximum single run time of just below 100 hours, this makes the system extremely flexible.

This manual will guide you through the requirements for setting up, operating and maintaining the system. It will facilitate the most efficient procedures to protect your product's integrity and ensure optimum performance at all times.

Safety

Safety Symbols

The following safety symbols are used throughout this manual and are defined as follows.



Hazards that can be harmful to health, or lead to serious damage or injury.



Possible risk to sample integrity.

Genevac Evaporators and Combustible Solvents



Please note it remains the responsibility of the user to consider safety when evaporating any combustible solvents, and to place the system in a well-ventilated environment. Genevac's position regarding evaporation of such solvents, particularly with respect to the European ATEX directive, is available on our website or from your local sales representative.

Electrical Safety



Important: the system must be earthed. This evaporation system is a safety class 1 product according to IEC classification. It must never be used with any interruption to the safety earth conductor. It is an installation category II product and is intended to operate from a normal, single-phase power supply.

This evaporator is designed for use in a degree 1 environment (no pollution, or only dry non-conductive pollution).

Any maintenance or repair of this product must be carried out by Genevac personnel (or approved representatives of Genevac) using only approved spare parts.

Limitations of Use



The **HT4-SII** and **HT4X-SII** evaporation systems are **unsuitable** for use under the following circumstances:

- With strong mineral acids such as HCl and HBr at all concentrations, unless specifically built to order.
- Evaporating diethyl ether and other similar low auto-ignition solvents.
- For use as a pressure vessel.

Options

Available options include:

- Bump protection (***Dri-Pure***™ and ***Variable Dri-Pure***).
- High power ***Coolheat*** lamps.
- Low UV lamp lenses.

Data Logging is available. The easy-to-use package provides the data needed for process and quality control.

Hardware Required:

- PC running 'Windows' (95, 98, NT, 2000 or XP).
- Around 5Mbytes of free space on your hard drive.
- Serial Port on the PC or USB to Serial Adapter.
- Serial Cable to connect the PC to the Evaporator.

Contact your sales representative for purchase and installation details.

Delivery and Installation

Installation and commissioning by Genevac personnel is an option available with every new Genevac HT system. Installation instructions are provided should the commissioning option not be selected.

Checking the Delivery

Check the contents of the delivery, against the delivery note, as soon as possible. Notify Genevac Ltd immediately if any parts are missing or damaged. (Refer to the back cover for contact details).

Arranging Commissioning

If your system is to be delivered separately, Genevac Ltd will contact you prior to delivery, to agree a convenient date to commission your system.

Training

Commissioning normally includes training in the basic operation of the system. Further in-house training is recommended to fully exploit the flexibility of the system.



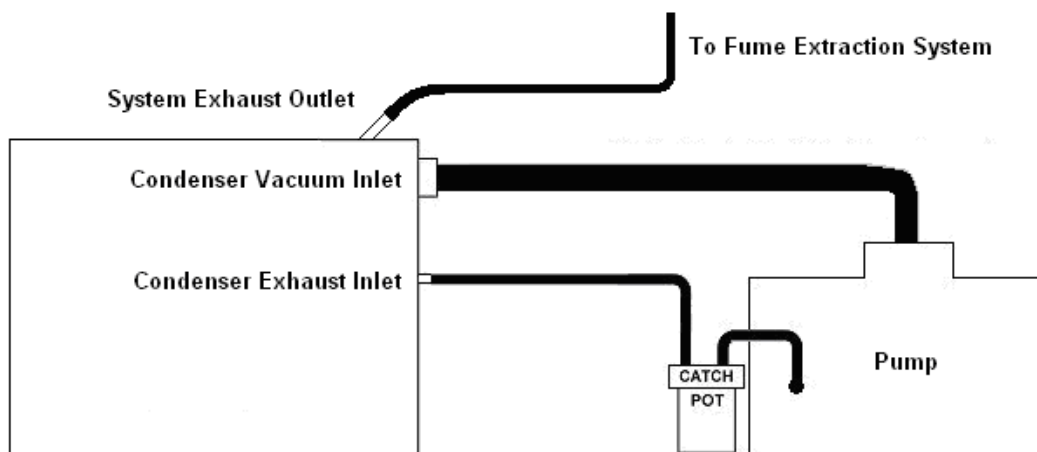
The HT4 evaporator must not be operated by personnel who lack the training or experience to comprehend the hazards that can arise when using the system. Personnel without such training require thorough instruction. These operating instructions should form the basis of this instruction.

Positioning the Evaporator



The evaporator must be sited on a level and sturdy work-surface. Position the evaporator at least 300mm away from the edge of a bench and the same distance clear of any breakable objects or areas where entrapment could occur. If this positioning requirement is impractical then the evaporator should be bolted to the bench or trolley, by its five mounting feet, using M10 high tensile bolts. Consult Genevac for advice on any other positioning requirements.

Pipe Connection Overview



Identifying the Parts

Control Cable Socket

HT4X-SII



Condenser Vacuum Inlet

Condenser Exhaust Inlet

HT4 Build Variants

Early build HT4X-SII



Pump Vacuum Inlet



HT4-SII

Pump Control Box

Mains Power Connector

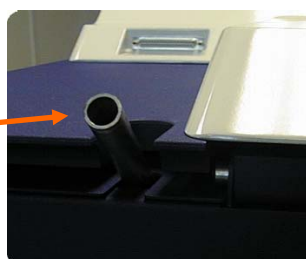


Catch-Pot Assembly

Pump Control Cable Socket

Pump Exhaust Outlet

System Exhaust Outlet



LyoSpeed System
Condenser Drain Manifold
with Single Drain Pipe



Chamber Drain Outlet

Condenser Drain Outlet

Exhaust Drain Outlet



Installing the System

These instructions refer to the installation of an **HT4X-SII** evaporator with a **scroll pump**. Details follow to show differences that may be encountered on different build variants.

Note: The power to the evaporator is delivered via the pump control cable.

Fit the **pump control box** to the scroll pump using the connectors, clamps and fasteners supplied. Fit the **pump exhaust outlet** and **catch-pot assembly** to the pump.

Place the evaporator and scroll pump in their intended location. Ensure there is enough space above the evaporator for the lid to open.

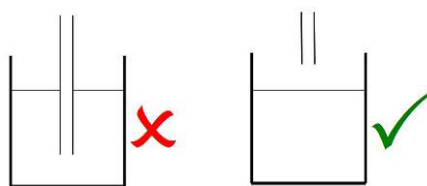
Connect the 16KF convoluted vacuum tube to the **pump vacuum inlet** via a 16KF to 25KF reducer, appropriate clamps and centre rings. Connect the other end of the convoluted vacuum tube to the **condenser vacuum inlet** on the back of the evaporator.

Connect a length of exhaust tube to the spigot on top of the **pump catch-pot**. Connect the other end of the exhaust tube to the **condenser exhaust inlet** on the back of the evaporator.

Connect the pump control cable to the **pump control cable socket**. Connect the other end of the control cable to the **control cable socket** on the back of the evaporator. **Note:** The pump control cable has a male connector at one end, a female at the other end and only connects to the corresponding sockets in the correct orientation. To connect the control cable, push the connector into the socket and twist the knurled grip clockwise until it clicks into place.

Connect the remaining length of exhaust tube to the **system exhaust outlet** and secure using a pipe clamp, connect the other end of the exhaust tube to the laboratory fume extraction system.

Connect drain pipes (not supplied) to all three drain connectors. Place the other end of the drain pipes in a suitable waste solvent container (not supplied). Ensure the end of the drain pipes cannot be submerged in the waste solvent.

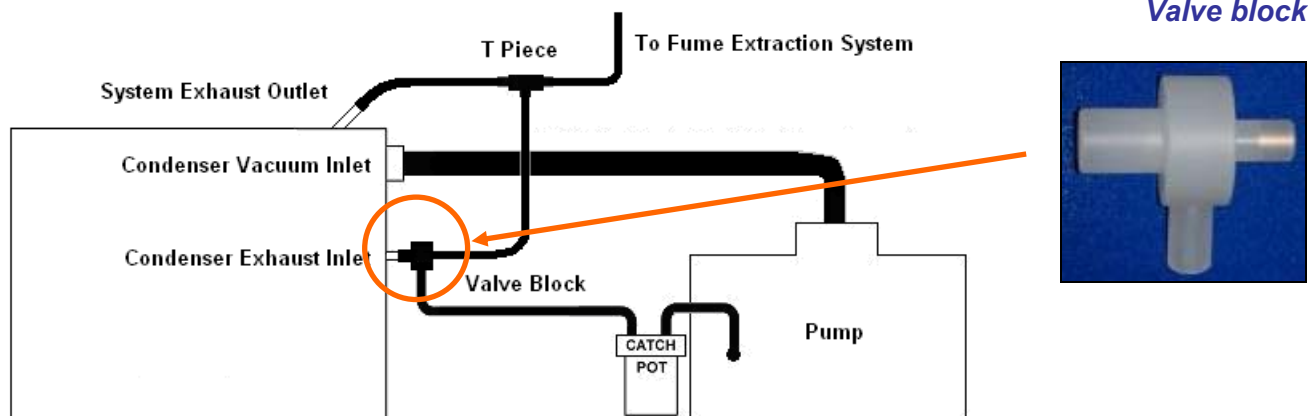


Connect the mains power cable to the pump **mains power connector**. Connect the mains power cable to a suitable mains power outlet.

The system is now ready to switch on. Carry out a safety inspection before operating the system.

Installation Variations: Valve Block

A valve block and T piece are required to connect a scroll pump to an **HT4-SII** or an early build variant **HT4X-SII** evaporator. These models can be recognised by metal condenser vacuum inlet and condenser exhaust inlet. The valve block is necessary to prevent the possibility of damaging back pressure being applied to the scroll pump exhaust if ice forms in the condenser exhaust pot (the later HT4X SII features an internal pressure relief valve).



Installation Variations: Lyospeed Systems

Connect the single solvent drain pipe to the drain outlet manifold of an HT4X SII **LyoSpeed** system.

Ensure the end of the drain pipe cannot be submerged in the waste solvent.

Balance pipe

Single drain



Installation Variations: CVP Vacuum Pump

CVP Pump exhaust outlet



CVP Pump vacuum inlet

Install the CVP pump in the same way as the scroll pump, the following additional instructions apply.

Connect a short length of exhaust tubing to the pump exhaust outlet and seal with a pipe clamp. Connect the other end of the short tube to a pump catch pot spigot and seal with a pipe clamp.

Connect another length of exhaust tubing to the second catch pot spigot, seal with a pipe clamp. Connect the other end of this exhaust tubing to the condenser exhaust inlet on the back of the evaporator.

Note: Early build versions of the CVP pump require a different pump control cable which connects to the pump via a plastic connector.

Do not attempt to exchange a CVP pump for a scroll pump. This operation requires a set-up in the system software that may only be carried out by Genevac Service or a trained representative of Genevac.

Safe Loading of Rotor – General Document



Genevac are obliged to include the following information in its entirety, irrespective of the system type.

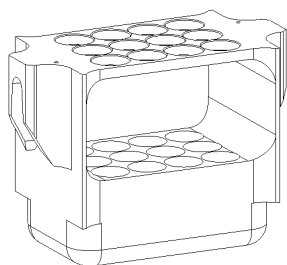
As with all centrifuges, Genevac centrifugal evaporators must be loaded correctly to remove the risk of damage. Failure to correctly load a system can lead to unrecoverable loss of samples and damage to the system. This guide is intended for new users and also as a reminder for more experienced users. The principles outlined apply to all Genevac evaporators.

The following instructions deal with various aspects of loading:

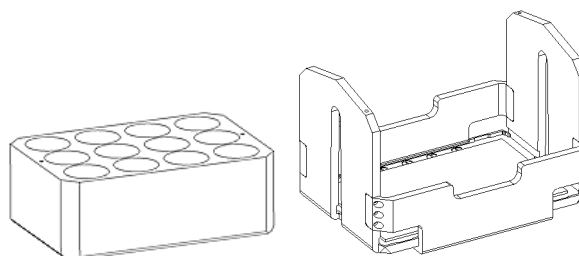
- Use of correct swings and sample holders
- Observation of weight limits
- Safe loading of sample holders into swings
- Balancing of swings and sample holders
- Good procedural practice

Nomenclature

Some Genevac systems feature **fixed rotors** which have angled holes for individual tubes or holders. The following instructions are primarily concerned with the more common **swung rotors**. The new range of Genevac sample holders (blue in colour) include integral swing holders such as the example shown.



Integral Swing / Tube Holder



Tube Holder and Side-Bridge Swing

Sample holders requiring a separate swing (or bucket) may also be used.

Each of these swing types lifts straight out of the rotor. The ideal swing type depends on the **sample holder** required. A large range of sample holders is available. Load the tubes, vials, beakers or flasks into the sample holders, then place the sample holders in the swings.

Use of Correct Accessories

Genevac supply a wide range of sample holders to cover a variety of plate, tube, vial or flask formats. Where no holder is available, custom units can be made. To ensure that bespoke holders are suitable and approved for use, new sample holders may be designed in collaboration with the Genevac R&D department.

Criteria for approval:-

- Mass within prescribed maximum limit.
- Matched sample holder mass within a set.
- Correct centre of gravity.
- Good thermal conductivity.
- Correct hole, form and size, tolerance to reduce risk of tube or vial breakage under centrifugal acceleration.

All Genevac holders are designed with these constraints in mind. 3rd party accessories may not be. With the exception of microtitre plates, do not load any non-Genevac holders into a system without gaining approval from Genevac. Failure to comply may result in unrecoverable loss of samples, severe damage to equipment and invalidation of the warranty.



All swings and holders must be approved for the Genevac system in which they are used (applicable particularly where Series I and Series II evaporators are used within the same laboratory). For example, some sample holders designed for use in Series II systems, might appear to fit in a Series I system, but would exceed the weight limitations. If in doubt, consult Genevac before using such holders.

There are also instances where a sample holder intended for one tube or vial, becomes unsuitable if used with something else, even though it might appear that the alternative tube fits. The **Genevac Accessories Brochure** indicates the maximum tube length that each holder is designed to take. Always adhere to these limits.

Similarly, sample holders intended for use in a Side-Bridge swing must not be used in an Open swing, even though they might appear to fit. The **Genevac Accessories Brochure** has a note: *For use in Side-Bridge Swing* by these items, but if in doubt, ask Genevac.

Please note that some (not all) of the *Bohdan Miniblock* system sample holders significantly exceed the mass limitations of a Genevac system. If you plan to use Bohdan Miniblocks in a Genevac system, please contact Genevac for a list of the relevant weights.

Adherence to Mass Limit

The total mass that can be loaded onto each position of a Genevac swung rotor. This mass includes:

- The swing
- The sample holder
- The tubes or vials
- The sample solutions

Do not exceed the mass limit under any circumstances. In most cases, with normal solvent volumes, sample holders sold by Genevac fall within the mass limit for a series II system. If in any doubt, load up a full swing and weigh it.

Safe Loading of Sample Holders into Swings



Sample Holder rotated



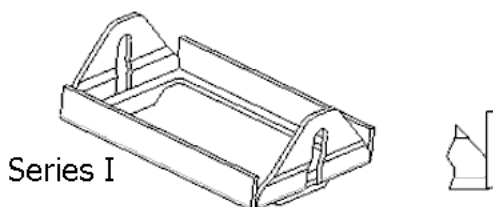
This information is applicable where Series 1 and series II equipment is used in the same laboratory.

There are two possible ways in which a sample holder may be miss-loaded into an older type of swing. One is to rotate the sample holder such that its corners rest on the edges of the swing. The other is to place the sample holder so that one side rests on the edge of the swing.

Both these modes of miss-loading are possible with a Series 1 *open* or *standard* swing, but are virtually impossible with a Series II swing. Care must still be taken when loading the samples.



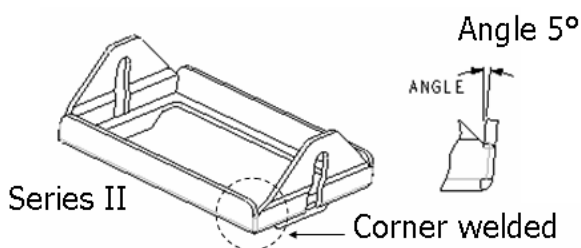
Sample Holder correctly seated



The Series 1 design is easy to distinguish. The corners are not welded.



Sample Holder on edge of swing



The Series II swing has angled sides and rounded corners that are welded.

Genevac offer an option to upgrade to the new range of sample holders and swings. This upgrade permanently avoids the possibility of mis-loading..



Sample Holder correctly seated

Balancing Sample Holders and Swings



Opposite pairs of swings must be balanced within 10g and the swings must be of the same type and version. Please note that there are several versions of **Side Bridge Swings** in circulation with a static weight ranging from 375g to 445g. Place swings of the same static weight, in diametric opposition on the rotor; ideally use the same version of swings in all four positions of each rotor level.

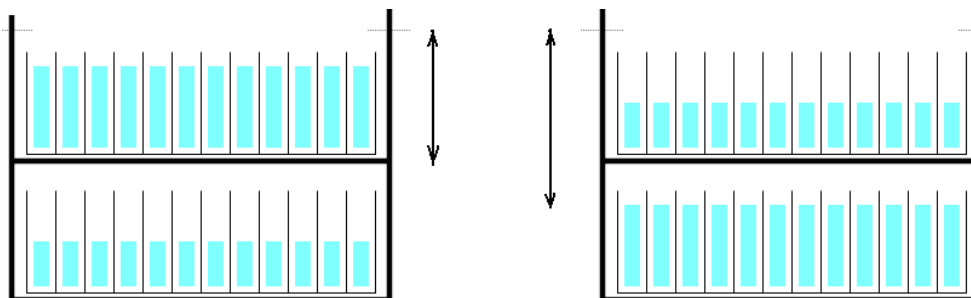
Genevac systems have some inbuilt tolerance for imbalance, and a safety system which stops the rotor if the *out-of-balance* is unacceptably high. However, to minimise noise nuisance and wear-and-tear the balance limit should always be observed.

If the evaporator is to be partially loaded, always fill the top rotor level first. Then fill the second level, then the third (third level applies to HT12-SII evaporators only).

Real loads may be balanced using *Dummy* samples. They must be of a similar solvent composition. For example, do not balance 200g of 50 / 50 - Water / Acetonitrile with 200g of water. Partway through the run, the acetonitrile evaporates but the water remains, resulting in an imbalance of 100g.

Note that with a system such as the **Fast-Stack™** swing, balancing is slightly more complex. Suppose a **Fast-Stack™ Deepwell** swing is used with two 96 well microtitre plates, 2ml per well. In one swing, the lower microtitre plate is empty, the upper microtitre plate is full, 1.8mls per well. On the other swing, the reverse is true.

The two swings now weigh the same. However, if placed opposite each other in on a rotor, would be imbalanced. This is because when the swings rotate to their operating attitude, the centre of mass of one is at a different radius to that of the other.



Swings are the same weight but not balanced

It is also preferable to run the evaporator with all four swings fitted to each level of the rotor. If there are only enough samples to fill two swings, it is better to distribute the samples into four holders (or, at least, place two empty holders in the rotor). This reduces the mechanical stress on the rotor and helps to distribute the heat flow evenly between the samples.

Good Procedural Practice

Finally, there are a few general rules for safe operation of a system:

- Only permit users, familiar with all the issues outlined in this document, to operate the equipment.
- Only load swings and sample holders that are approved by Genevac.
- The same user should be responsible for loading and starting the system.
- Never leave the system unevenly loaded with the door closed. Someone may start it.
- Never start, or restart a system without checking it is evenly loaded, all sample holders are correctly seated and all holders swing freely.



Miss-loading may result in unrecoverable sample loss, damage to equipment, and could void the warranty.

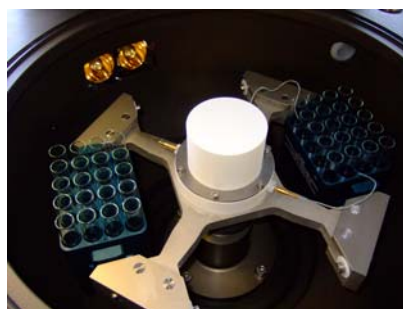
Loading the Rotor

Samples in the chamber are subjected to accelerations of up to 500G. The maximum load capacity is 1.5kg per swing. The load capacity includes tubes, solvent, sample, sample holder and swing.



Always observe the following Precautions

- Never exceed the maximum load capacity of 1.5 kg per swing.
- Ensure tubes locate correctly in tube holders.
- Locate sample blocks correctly in sample swings.
- Load two or four tube holders in opposite and balanced configurations.
- Distribute tubes in sample holders symmetrically.
- Balance oppositely loaded pairs of sample holders to within 10g.



Rotate the rotor by hand. Check that all tube holders and plates are correctly located and swing freely. Use only the sample holders supplied with the system, or that are specified for use with the system. Never use non Genevac approved sample holders.

Genevac Ltd will not accept responsibility for any loss or damage incurred by improperly or excessively loaded rotors.

Solvents

Common Acceptable Solvents

Solvent	Density (g/cm ³)	Density Temp. (°C)	Autoprogram Available
Acetic Acid	1.05	20	N
Acetonitrile (ACN)	0.79	20	Y
Ammonium Hydroxide (NH ₃ OH)	0.90	20	Y
Chloroform (TCM)	1.48	20	Y
Dichloroethane (DCE)	1.24	20	Y
Dichloromethane (DCM)	1.33	20	Y
Dimethylacetamide (DMAc)	0.94	20	Y
Dimethylamine (DMA)	0.68	20	N
Dimethylformamide (DMF)	0.94	25	Y
Dimethylsulfoxide (DMSO)	1.10	20	Y
Ethanol (EtOH)	0.79	20	Y
Ethyl Acetate (EtAc)	0.90	20	Y
Hexane (Hex)	0.65	20	Y
Isopropanol	0.79	20	N
Methanol (MeOH)	0.79	20	Y
N-Methyl-2-pyrrolidone (NMP)	1.02	25	Y
Pyridine	0.98	20	N
Tetrahydrofuran (THF)	0.89	20	Y
Toluene (PhMe)	0.87	20	Y
Tri Ethyl Amine (TEA)	0.73	20	Y
Trifluoroacetic acid (TFA)	1.54	20	Y
Water	1.00	20	Y

HCl, Thionyl Chloride or Di Ethyl Ether compatibilities are not available.

Genevac and the ATEX Directive



Please note that it remains the responsibility of the user to consider any solvents being evaporated within the context of the ATEX directive. The presence of solvents on the list above indicates only that they will not damage the system. If further information is required, please contact your Sales Representative or visit <http://www.genevac.com/>

Those solvents with a **Y** in the **Autoprogram Available** column are available from the **Solvent** drop down list when setting up a run. The solvents with an **N** against them must be entered using the **Other** option and the rest of the data entered manually

Getting Started

The following notes provide basic instructions for starting up the evaporation system. Refer to the relevant sections of this manual for detailed information on specific steps.

Switching on the Pump

Connect the pump to the mains and switch on the mains.

Switch on the pump power switch.

Note: The pump does not start until the evaporator is switched on.



CVP pump



Pump Power Switches

Switching on the Evaporator

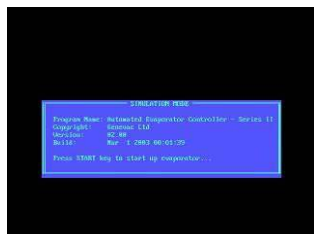
Switch on the evaporator power switch.

There is a short delay while the imbedded PC completes its boot up sequence. The display shows the **Software Control** screen.

The system is now in stand-by mode.



Evaporator power switch



Software Control Screen

Press the **START** key  to start the system:-

- The pump starts.
- The display shows the **Run Select** screen.
- The condenser starts after a delay of approximately three minutes.

Note: The system is not available for use until:

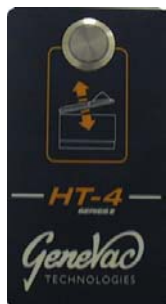
- The pump reaches its operating temperature (CVP pumps only).
- The system **Pump Warming** delay times-out (HCL systems with a scroll pump only).
- The condenser temperature cools below 0°C.

Opening the Lid

Press the button on the front panel to open, stop and close the lid.

The lid is operated by an electric actuator, and is controlled by safety sensors. If an object prevents the lid closing, it re-opens automatically to prevent entrapment.

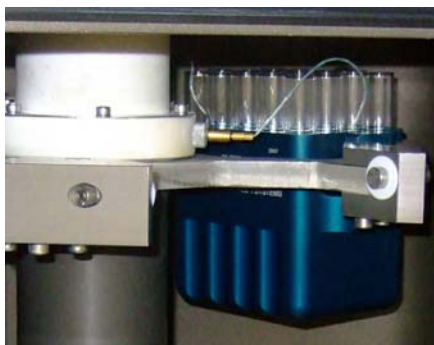
Note: The lid only opens if the rotor is stationary and the chamber is at atmospheric pressure.



Lid Open Button

Using SampleGuard™

SampleGuard Transmitter and Probe



Sample holder probe hole



SampleGuard is a dual channel temperature monitoring system that operates when the rotor spins. One channel allows the evaporator to control the temperature of a sample holder, the other monitors the temperature of a sample within the holder.

The temperature differential, caused by evaporative cooling, is used to monitor sample drying progress.

Connect the **SampleGuard Probes** to the **SampleGuard Transmitter** as shown. The numbers **1** and **2**, stamped into the SampleGuard transmitter, identify the input connectors for each channel.

Connect a **SampleGuard Stuffer Plug** to the third connector for normal system operation.

Insert the **Channel 1** (control channel) temperature probe into the small probe hole in one of the sample holders.

Insert the **Channel 2** temperature probe into a sample tube / vial / well etc.

Press the **SETTINGS** key to display the **Run Data** screen.

Use the cursor keys to select the **SampleGuard Control Channel** field and select channel 1 as the control channel.

Note: Only use the option to select channel 2 as the **SampleGuard Control Channel** if there is a fault with channel 1 or its associated temperature probe. In this instance, insert the channel 2 probe into the probe hole of a sample holder. The sample temperature cannot now be monitored.

Use the cursor keys to select the **SampleGuard Control Temperature** field and press **ENTER**.

Input the new value and press **ENTER** again.

The system requests a confirmation command to make the change and close the **Run Data** screen. Press **Y** for yes and **N** for no.

The SampleGuard transmitter requires the rotor to spin at sufficient speed before it operates. The message **TxD** displayed on the screen, indicates that the SampleGuard is not transmitting. This could be because the rotor is spinning up or is running at **Very Low** speed.

Note: Significant cooling (and possibly freezing) occurs during evaporation (the extent is dependant upon the solvent). It is advisable to position the sample probe in a sample near the centre of the holder. Since the central wells are surrounded by other frozen or cold wells, there is a significant thermal drain during evaporation.

This effect is particularly prevalent when using microtitre plates. Under these conditions, dryness may occur in the outer wells whilst the central samples remain in solution or frozen. The use of Genevac Heat Transfer Plates is beneficial in reducing this effect, by providing an even heat transfer across the plate.

When positioning the sample probe, always ensure that the probe tip is located firmly at the bottom of the well, vial or tube. Once positioned, the probe stays firmly in position under centrifugal force.

Verifying the Sample Guard Calibration



Sample Guard Stuffer Plug



Access temperature display with Hidden Key



Comparison Thermometer

The following operation requires the use of a **Sample Guard Power supply**. This is may be ordered as an option or may be purchased from Genevac.

With the evaporator switched on, open the evaporator door and remove the **Sample Guard Stuffer Plug**.

Connect SampleGuard probes to channels 1 and 2 and immerse their ends in a beaker of water at approximately 40°C.

Connect the power lead from the Sample Guard Power Supply to connector 3 of the Sample Guard transmitter. Plug the Sample Guard Power Supply into a suitable mains power outlet and switch it on.

Close the door so that the screen may be viewed, but do not lock it. This prevents any chance of inadvertently starting the rotor with the power supply cable connected.

Press a hidden key on the keypad (as shown) and verify the indicated temperatures against a calibrated digital thermometer.

Compare the readings on the display with that of a calibrated reference thermometer measuring the temperature of the same water.

The frequency of verification and record retention, should meet the requirements of your ISO/EN/BS 9000 series or NAMAS procedures.

Disconnect the SampleGuard Power Supply lead from the SampleGuard Transmitter, refit the SampleGuard Stuffer Plug.

The SampleGuard system will not operate without the Stuffer Plug fitted.

The Condenser

The condenser starts automatically a short time (approximately 3 minutes) after the evaporator is switched on. There is a further short delay before the condenser reaches operating temperature. Drain the condenser (defrost first if necessary) before each run and at the end of each day.

Access the **DEFROST / DRAIN CYCLE** for the condenser via the **Select Run Screen**. See the section entitled: **Select Run Screen** for details.

Manual Defrost and Drain

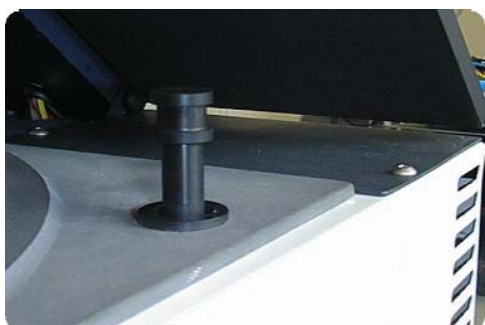
To start the defrost cycle, move the cursor to the **DEFROST / DRAIN CYCLE** using the up-down cursor keys and press the **ENTER** key.

The **DEFROST VALVE** opens and the defrost cycle begins.

The required frequency of defrosting and draining the condenser depends on the volume and nature of the evaporated solvents.

The maximum defrost period should be no longer than two hours. Defrost and drain the condenser after each run to obtain optimum performance from the condenser.

Open the lid and lift the drain valve plunger to open the valves.



Note: Close the drain valves manually, do not rely on the lid to push down the drain valve plunger when it closes.



Before opening the condenser drain valves, ensure that pipes connect the **chamber drain**, **condenser drain** and **exhaust drain** outlets (or single drain pipe, for **LyoSpeed** systems) to a suitable waste solvent container.

Auto Defrost and Drain (LyoSpeed enabled Systems Only)



Ensure that a suitable waste solvent container is connected to the condenser drain pipe at all times. Draining occurs automatically on **LyoSpeed** systems, even after the run has completed. Ensure that the end of the drain hose remains above the level of waste solvent in the container.

Flushing the Condenser

Use the condenser flushing system (if fitted) to clean traces of volatile solvent from the condenser before evaporating a less volatile solvents, the condenser operates most efficiently when it is drained and clean.

Always drain the condenser before using the condenser flusher. Select **FLUSH** on the HT4-SII **Menu Bar** and press the **START** key. Wait until the system vacuums down to approximately 5mabr. Pour approximately 100 ml of acetone into the flushing solvent container and open the **flushing valve**.



Close the flushing valve after use. Stop the system and allow it to vent to atmospheric pressure from the condenser vacuum pot. Repeat the flushing procedure as necessary.

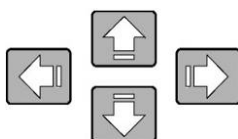
Never use the flushing valve to vent the system,

Evaporator Controls

The evaporator keyboard, controls and display screens, are designed for ease and simplicity of use. The frequently used function keys are on the left hand side of the keyboard. The right side illuminates to accept input when the **Run data** screen is accessed.



Press the **START** and **STOP** keys to start and stop the selected run.



Press the **Cursor Direction Keys** to move around the screen in the direction of the arrows.



Press the **Enter** key to open or close fields in the **Run Data** screen for inputting or amending data. **Note:** the **START** key acts as an enter key when the keyboard is not illuminated.



Press the **Menu** key to display the **User Menu** screen. This only operates when the rotor is not spinning and the chamber is at atmospheric pressure.



Press the **Display** key to show the graph from the last completed run. This function only operates when a run has been completed. Press the **Cursor Direction Keys** to show different segments of the graph.



Press the **Settings** key to display the **Run Data** screen for the selected run. **Note:** the **Run Data** screen can also be accessed from the **View / Edit Details** option on the **SELECT RUN** screen.

Control Screens: an Overview

There are three main control screens. The information on this page is a brief overview of what the screens do. Detailed descriptions are provided in following sections.

1. Select Run Screen.

SELECT RUN					
No.	Name	Solvent	Vol.	Holder	T. Nxt
1	Oligosynthesis	Ammonia	2ml	DEEP WE	35 0
2	Oligo Purification	Water/ACN	0ml	DEEP WE	38 0
3	PCR Purification	Water/ACN	1ml	DEEP WE	38 0
4	RNA Purification	Water	10ml	24 X 15	4 0
5	Oligo Production	Aqueous Mixture	4ml	58 ml I	48 0
6					
7					
8					
9					
10					
GENEVAC LTD					
MENU					
START EDIT GRAPH CLEAR COPY TO DRAIN DEFROST FLUSH OPTIONS					
HELP					
Select Run with ↑↓, Menu Item with ←→ keys, press START to execute					
Starts selected Run using parameters stored					

The **Select Run** screen has the capacity to store up to 100 different run profiles. Runs 51 to 98 are preloaded to provide a range of standard run configurations. Runs 99 and 100 are for diagnostic use by engineers. Run locations 1 to 50 are available for users to insert their own run profiles.

The **Select Run** screen is displayed shortly after the system is switched on. The most commonly used run is highlighted by a white band across the screen.

2. Run Data Screen

Run No. 91 — Last Used (Not yet used) — Last Changed (New entry)	
Run Data Protection	No
Next Run Number (0 for none)	0
Run Name	
Sample Holder Type	
Sample Volume (ml)	0.0
Solvent	
IR Sensor Control Temperature [°C]	20
Cool/Heat Enable Pressure (mbar)	100
Chamber Temperature Control [°C]	Wait to cool to 20
Rotor Speed	High
Heat-Off Option	Elapsed Time
Heat-Off Elapsed Time (HH:MM)	00:00
End-of-Run Option	Elapsed Time
End-of-Run Elapsed Time (HH:MM)	00:00
End of Run Defrost and Drain	No
Pressure Control Regime	Dri-Pure
Final Controlled Pressure (mbar)	0.0
GENEVAC LTD	
HELP	
↑↓ to select, ENTER to edit, MENU/ESC to exit.	
Select level of Run Data protection	

Press the **Settings** key to display the **Run Data** screen. It shows the parameters that combine to form a run profile. The screen is used to create and edit runs. It may be accessed whilst a run is in progress, allowing control parameters to be adjusted mid-run.

3. Run Log Screen

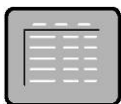


The **Run Log** screen is displayed whilst a run is in progress. It shows the current status of the evaporator in numerical and graphical form. When the SampleGuard probes are appropriately configured, it is possible to extrapolate information from the graph that relates to sample drying progress. An evaporative cooling effect can be seen in the divergence of the channel 1 and channel 2 traces. Conversely, converging traces reveal when a sample is dry. See **Using SampleGuard** for details.

Select Run Screen

The **Select Run** screen lists a library of up to 100 evaporation run profiles. Unused run locations remain blank and are available for creating new runs. Press the **Cursor Direction** up and down keys to scroll through the run profiles. Run profiles are displayed on the screen 10 at a time.

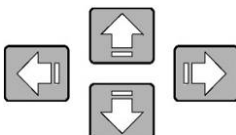
SELECT RUN						
No.	Name	Solvent	Vol.	Holder	T.	Nxt
1	Oligosynthesis	Ammonia	2ml	DEEP WE	35	0
2	Oligo Purification	Water/ACN	0ml	DEEP WE	30	0
3	PCR Purification	Water/ACN	1ml	DEEP WE	30	0
4	RNA Purification	Water	10ml	24 X 15	4	0
5	Oligo Production	Aqueous Mixture	4ml	50 ml T	40	0
6						
7						
8						
9						
10						
GENEVAC LTD						
MENU						
START EDIT GRAPH CLEAR COPY TO DRAIN DEFROST FLUSH OPTIONS						
HELP						
Select Run with ↑↓, Menu Item with ←→ keys, press START to execute						
Starts selected Run using parameters stored						



Press the **Settings** key to enter the **Run Data** screen (see the section entitled: **Run Data Screen** for details).

START

Press the **START** key to execute the highlighted option. **Note:** the **Run Log** screen is shown when the run starts.




Press the **cursor direction** left and right keys to highlight the options on the horizontal menu bar near the bottom of the screen. Press the up and down keys move the **white band** cursor.

Note: The default position for the horizontal menu bar cursor is with **START** highlighted. Press the **cursor direction** left and right keys to return the cursor to **START** after using any of the other menu options.

Functions of the Menu Bar



- **START:** Starts the highlighted run.
- **EDIT:** Alternative route to the **Run Data** screen.
- **GRAPH:** Alternative route to the graph displayed by the display key .
- **CLEAR:** Clears the highlighted run profile.
- **COPY TO:** The highlighted run profile may be copied to a different location. This feature is useful if several similar run profiles need to be created. Copy and edit a profile instead of creating each one from scratch.
- **DRAIN:** Drains the condenser. Applicable only to **LyoSpeed** or automated systems.
- **DEFROST:** Enters the **Condenser Defrost** menu.
- **FLUSH:** Enters the **Condenser Flush** menu. Applicable only if the flush option is installed.
- **OPTIONS:** Enters the **Options Menu**.

Entering Control Data

Enter data in the **Run Data** screen.

From start up of the system and the software control screen, press **START** to display the **Select Run screen**.



Select the **View / Edit Details** option and press **START** to display the **Run Data** screen.



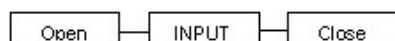
Use the **UP** and **DOWN** cursor keys to move between fields.



Unless otherwise stated, press **ENTER** to open a field,



Input your requirement and press **ENTER** again to close the field.



Run Data Screen

Each of the method profiles shown on the **Select Run** screen is generated from the **Run Data** screen. Select the required run profile and press the **Settings** key to enter the Run Data screen. The parameters of the selected run are shown on the screen. A default set of parameters is shown if an unused run location is selected. The Run Data screen can also be accessed when a run is in progress.

```

Run No. 6 — Last Used [Not yet used] — Last Changed [New entry]
Run Data Locked No
Next Run Number [0 for none] 0
Run Name
Solvent
Sample Holder Type
Sample Volume 0.00

SampleGuard Control Temperature [°C] 35
SampleGuard Control Channel 1
CoolHeat Enable Pressure [mbar] 100
Minimum Chamber Temperature [°C] 35
Rotor Speed [High/Low] Low

Heat-Off Option Elapsed Time
Heat-Off Elapsed Time [HH:MM] 00:00
End-of-Run Option Elapsed Time
End-of-Run Elapsed Time [HH:MM] 00:00

Pressure Control Regime Controlled Pressure
Controlled Pressure [mbar] 0

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HELP
↑↓ to select, ENTER to edit. MENU/ESC to exit.
Toggle "Lock" protection of Run Data

```

Press the **cursor direction** up or down keys to move between fields. Press the **Enter** key to open a field. Enter a new value for the relevant parameter and press the **Enter** key again to close the field.

Run Parameter Details

Run Data Protection

The run profile may be protected against accidental alteration. Set the **Run Data Protection** field to **No** before attempting to edit an existing run profile. Set the **Run Data Protection** field to **Yes** to lock the profile.

Next Run Number

Runs may be linked together. Enter the number of the run that requires to be linked. The linked run commences when the current run finishes. Enter 0 in the **Next Run Number** field if no run is to be linked.

Note: A run may be linked to itself. This causes the run to continue repeatedly.

Run Name

Enter a name (no more than 20 characters) to identify the run profile. Text entered on the keyboard appears in the bottom part of the screen. Press the **Enter** key to enter the text into the **Run Name** field.

Sample Holder Type

```

Run No. 7 — Last Used [Not yet used] — Last Changed [New entry] —
Run Data Locked
Next Run Number [0 for none]
Run Name
Solvent
Sample Holder Type
Sample Volume

SampleGuard Control Temperature [°C]
SampleGuard Control Channel
CoolHeat Enable Pressure [mbar]
Minimum Chamber Temperature [°C]
Rotor Speed
Heat-Off Option
Heat-Off Elapsed Time [HH:MM]
End-of-Run Option
End-of-Run Elapsed Time [HH:MM]
Pressure Control Regime
Dri-Pure Final Controlled Pressure [mbar]

SAMPLE HOLDER
Select Sample Holder
Microtitre Plate
Deep Well Plate
Tube/Vial Holder
Other

High
Elapsed Time 00:00
Elapsed Time 00:00
Dri-Pure 0

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HELP
↑↓ to select, ENTER to accept, ESC to cancel.
Sample holder type to be entered

```

Select a sample holder type from the window that opens. If the required sample holder option is not available, select *other* and enter the details (no more than 31 characters).

Sample Volume

Enter a value for the volume of sample in each individual well / tube / vial.

Solvent

```

Run No. 91 — Last Used [Not yet used] —
Run Data Locked
Next Run Number [0 for none]
Run Name
Solvent
Sample Holder Type
Sample Volume

SampleGuard Control Temperature [°C]
SampleGuard Control Channel
CoolHeat Enable Pressure [mbar]
Minimum Chamber Temperature [°C]
Rotor Speed
Heat-Off Option
Heat-Off Elapsed Time [HH:MM]
End-of-Run Option
End-of-Run Elapsed Time [HH:MM]
Pressure Control Regime
Dri-Pure Final Controlled Pressure [mbar]

SOLVENT
Acetonitrile (ACN)
Ammonium Hydroxide (NH3OH)
Chloroform (TCE)
DCM
Diethyl Ether (DEE)
DMF
DMI
DMSO
Ethanol (EtOH)
Ethyl Acetate (EtAc)
Hexane (Hex)
Methanol (MeOH)
NMP
TFA
THF
Toluene (PhMe)
Water (H2O)
Other
Clear
Done

GENEVAC LTD
HELP
↑↓/ENTER to select/deselect, 'Done'/ENTER to accept, ESC to cancel.

```

Select a solvent from the window that opens. More than one solvent may be selected if using a mixture of solvents. Selected solvents are highlighted in blue. Select **Done** when the solvent or solvents have been selected. Alternatively, select **Other** and enter the solvent name (no more than 31 characters).

Auto Program

At this point, the option to select **Auto Program** is presented.

```

Run No. 91 — Last Used [Not yet used] — SOLVENT
Run Data Locked
Next Run Number [0 for none]
Run Name
Sample Holder Type
Sample Volume [ml]
SampleGuard Control Temperature [°C]
SampleGuard Control Channel
CoolHeat Enable Pressure [mbar]
Chamber Temperature Control [°C]
Rotor Speed

Heat-Off Option
Heat-Off Elapsed Time [HH:MM]
End-of-Run Option
End-of-Run Elapsed Time [HH:MM]

Pressure Control Regime
Dri-Pure Final Controlled Pressure [mbar]

Auto Programme remaining parameters? (Yes/No)

Acetonitrile (ACN)
Ammonium Hydroxide (NH3OH)
Chloroform (TCE)
DCM
Diethyl Ether (DEE)
DMF
DMI
DMSO
Ethanol (EtOH)
Ethyl Acetate (EtAc)
Hexane (Hex)
Methanol (MeOH)
NMP
TFA
THF
Toluene (PhMe)
Water (H2O)
Other
Clear
Done

GENEVAC LTD
HELP
↑↓/ENTER to select/deselect, select Done when finished, ESC to cancel.
EtAc+Hex
  
```

Select **Yes** to allow the evaporator to set the remaining run parameters or **No** to continue entering the remaining parameter field manually.

```

Run No. 91 — Last Used 14:47 14/07/03 — Last Changed 14:47 14/07/03
Run Data Protection No
Next Run Number [0 for none] 0
Run Name Example
Sample Holder Type Deep Well Plate
Sample Volume [ml] 0.7
Solvent EtAc+Hex

SampleGuard Control Temperature [°C] 40
SampleGuard Control Channel 1
CoolHeat Enable Pressure [mbar] 200
Chamber Temperature Control [°C] AUTO: Cool to 32, Control at 20
Rotor Speed Low

Heat-Off Option Heat Flow
Heat-Off Threshold Not Applicable
End-of-Run Option Not Applicable
End-of-Run Threshold Not Applicable
End of Run Defrost and Drain Yes
Pressure Control Regime Controlled Pressure
Controlled Pressure [mbar] 9.5

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HELP
↑↓ to select, ENTER to edit. MENU/ESC to exit.
Temperature to maintain using SampleGuard
  
```

The screen shows the automatically programmed run parameters in blue. These parameters may be overwritten manually. Manually overwritten parameters are shown in white.

Note: There are a couple of restrictions to bear in mind when using the **Auto Program** function:

- **Auto Program** only works with the commonly used solvents shown in the **Solvent** window.
- If more than one solvent is selected, the evaporator selects a run profile which is suitable for the highest boiling point solvent. However, this may not be the most efficient method for the mixture. It is usually more efficient to set a run profile for the more volatile solvent, and link this to another run profile for the other component (or components) of the mixture.

The following details relate to the remaining run profile fields when the **Auto Programme** function is not used.

SampleGuard Control Temperature

Enter a temperature value between -20 and +70°C. The system heats the sample holders to this temperature. **Note:** For evaporators equipped with infra red temperature control, set **IR Control Temperature** instead of SampleGuard Control Temperature.



Only select control temperatures that are safe for the dried sample.

SampleGuard Control Channel

The default setting is channel 1. The option to change to channel 2 is provided to allow continued operation of the system should a fault arise with channel 1 or its associated probe.

CoolHeat Enable Pressure

Enter a pressure value between 5 and 400mbar. The infra red lamps do not come on until the CoolHeat Enable Pressure is reached. This prevents sudden uncontrolled boiling of volatile solvents as the chamber pressure reduces.

Chamber Temperature Control

```

Run No. 91 — Last Used 12:25 03/03/03 — Last Changed 12:31 03/03/03 —
Run Data Locked No
Next Run Number [0 for none] 0
Run Name Example
Sample Holder Type Deep Well Plate
Sample Volume [ml] 0.70
Solvent EtAc+Hex

SampleGuard Control Temperature [
SampleGuard Control Channel
CoolHeat Enable Pressure [mbar]
Chamber Temperature Control [°C]
Rotor Speed

Heat-Off Option
Heat-Off Threshold
End-of-Run Option
End-of-Run Threshold Not Applicable
End of Run Defrost and Drain Yes
Pressure Control Regime Controlled Pressure
Controlled Pressure [mbar] 10

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HELP
↑↓ to select, ENTER to accept, ESC to cancel.
Chamber temperature controlled to suit selected solvent or mixture
  
```

Select from the options that appear in the window and enter a temperature value between 0 and 45°C.

Use **Wait for Chamber to Heat** to pre-heat the vacuum chamber before removing high boiling point solvents. The system heats the chamber to the set value before allowing the run to start. This prevents high boiling point solvents condensing on the chamber walls. **Note:** The chamber heaters take approximately a minute to raise the chamber temperature by 1°C.

Use **Wait for Chamber to Cool** when removing volatile solvents or mixtures prone to bumping. The system waits for the chamber to cool before allowing the run to start. This helps to prevent sudden uncontrolled boiling as the chamber pressure reduces. **Note:** The chamber will not cool below the ambient temperature.

Use **Automatic Control** to slightly speed up the evaporation of high boiling point solvents. The system continues to heat the chamber temperature to the set value while the run commences.

Rotor Speed

```

Run No. 91 — Last Used 12:25 03/03/03 — L — ROTOR SPEED — 03
Run Data Locked
Next Run Number [0 for none]
Run Name
Sample Holder Type
Sample Volume [ml]
Solvent

SampleGuard Control Temperature [°C]
SampleGuard Control Channel
CoolHeat Enable Pressure [mbar]
Chamber Temperature Control [°C]      AUTO: Cool to 32, Control at 20
Rotor Speed                            Low

Heat-Off Option                        Heat Flow
Heat-Off Threshold                    Not Applicable
End-of-Run Option                    Not Applicable
End-of-Run Threshold                  Not Applicable
End of Run Defrost and Drain          Yes
Pressure Control Regime                Controlled Pressure
Controlled Pressure [mbar]            10

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HELP
↑↓ to select, ENTER to accept, ESC to cancel.
Rotor Speed Low

```

Select one of the following rotor speed options:

High: If microtitre plates or very full tubes are to be used, or if the solvent (or solvent mixtures) are prone to bumping. Samples are subjected to an acceleration force approximately equal to 500G.

Low: for most other applications.

There are few applications where **very low** speed is required. The option is designed to prevent the compression of dried compound during lyophilisation. However, experimentation reveals the effect to be negligible in most circumstances. **Note:** The SampleGuard system does not function when the **Very Low** rotor speed is selected. Therefore, heat input is not possible via the infra red lamps.

Heat Off Option

```

Run No. 91 — Last Used 14:40 03/03/03 — Last Changed 12:35 03/03/03 —
Run Data Locked                               No
Next Run Number [0 for none]                   0
Run Name                                       Example
Sample Holder Type                           Deep Well Plate
Sample Volume [ml]                             0.70
Solvent                                       EtAc+Hex

SampleGuard Control Temperature
SampleGuard Control Channel
CoolHeat Enable Pressure [mbar]
Chamber Temperature Control [°C]
Rotor Speed

HEAT-OFF OPTION
Select option for turning off CoolHeat
Elapsed Time
Heat Flow
Sample Detection

Heat-Off Option
Heat-Off Threshold
End-of-Run Option
End-of-Run Threshold
End of Run Defrost and Drain                  Yes
Pressure Control Regime                      Controlled Pressure
Controlled Pressure [mbar]                    10

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HELP
↑↓ to select, ENTER to accept, ESC to cancel.
Switch CoolHeat off when Heat Flow drops below predetermined level

```

The **Heat Off Option** may be set independently from the **End of Run** option. This allows a final drying stage to run without heat input from the IR lamps.

Select one of the following options for **End of Run** and / or **Heat Off**.

Elapsed Time

Enter independent times for heat off and for end of run. The evaporator turns off the IR lamps when the heat off time elapses, and allows the system to run without heat input until the end of run time elapses. This feature enables the protection of thermally sensitive samples as they approach dryness, while ensuring that all samples are dried completely.

Heat Flow

The system monitors the rate of evaporation by looking at the demand for heat input that is required to maintain the SampleGuard control temperature. When sample dryness is detected, the system turns off the IR lamps, and allows a final drying stage before stopping the run.

Sample Detection

The evaporator monitors the sample temperature. It detects dryness when evaporative cooling ceases, turns off the IR lamps and then stops the run. **Note:** Two temperature probes are required for **Sample Detection**. See **Using the SampleGuard** for details.

Note: Evaporators with IR temperature control, have no sample guard transmitter and do not offer the **Sample Detection** option for end of run detection. Set the **IR Control Temperature** in the same way as the **SampleGuard Control Temperature**.

End of Run Defrost and Drain

Select yes to defrost and drain condenser at the end of the stage. See the section entitled: **Getting the Best From Auto Defrost and Drain**.

Pressure Control Regime

Select one of the following:

Full Vacuum: The evaporator reduces the chamber to the lowest attainable pressure.

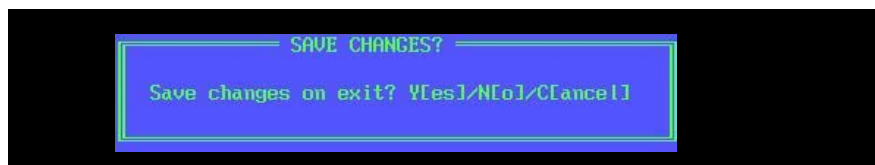
Controlled Pressure: Enter a value for the required chamber pressure.

DriPure: The evaporator selects high rotor speed and reduces the chamber pressure slowly. Use this setting if bumping is anticipated.

VariableDriPure: Enter values for **Dri-Pure Start Pressure**, **Dri-Pure End Pressure**, **Dri-Pure Ramp Time** and **Final Controlled Pressure**. This offers a more flexibility than the standard **Dri-Pure** option. The evaporator selects high rotor speed and reduces the pressure according to the set parameters.

To Save the settings and Create a new Run Profile

Press the **ESC** key on the keyboard. Press the **Y** key to confirm the changes.



Run Log Screen

The **Run Log** screen shows information relating to sample drying progress. Relevant system status information is shown with key parameter values represented graphically to reveal change trends in real time.

Numerical values are shown for:

- **Channel 1 Temperature:** *SampleGuard* channel 1, probe (connected to sample holder).
- **Channel 2 Temperature:** *SampleGuard* channel 2, optional, probes (submerged in sample).
- **Chamber Temperature:** Temperature of vacuum chamber wall.
- **Condenser Gas Temperature:** Temperature of coolant gas entering condenser.
- **Pressure:** Vacuum chamber (and condenser) pressure.
- **Spin Rate:** Rotor speed.
- **Total:** Time elapsed since run started, including completed linked stages.
- **This Stage:** Time elapsed since start of current stage, changes to **End of Run** or **End of Stage** and begins to count down when final run time is known.

Graphical display for:

- **Channel 1 Temperature**
- **Channel 2 Temperature**
- **Chamber Pressure:** Logarithmic scale.
- **Spin rate**

Other relevant system status appears above and below the graph area.

Alarms

If an error is encountered, a description of the problem is shown in the **ALARMS** field. Safety critical errors cause the evaporator to stop and report the reason for stoppage, non critical errors allow the evaporator to continue with compromised performance, and alert the user to the fault. **Note:** Rectify the problem and switch the evaporator off to clear the error from the screen.



Press the **DISPLAY** key during a run to return to the **RUN LOG** screen.

Press the **STOP** to end the run at any time.

The following screen appears after the run finishes.

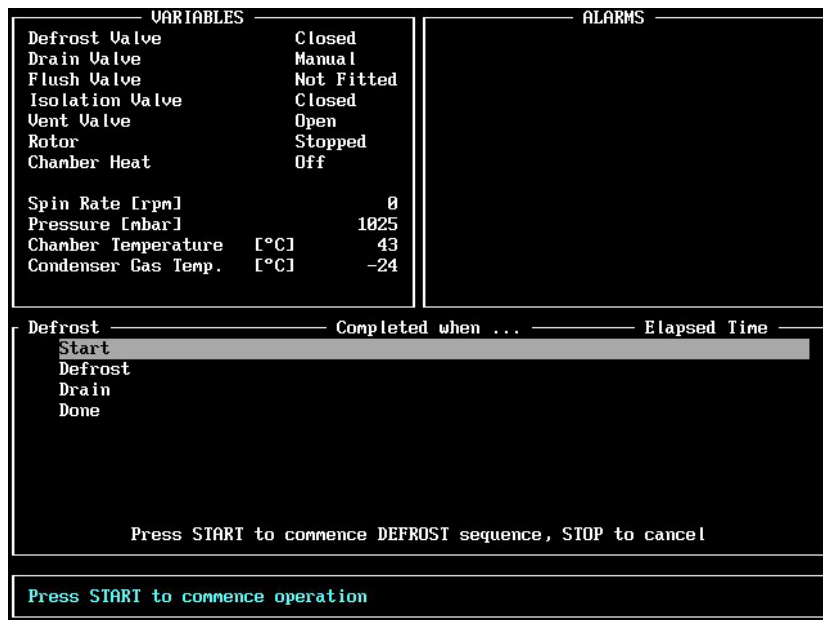


Select **Skip**, **Drain** or **Defrost** using the numeric keys indicated.

Skip: The evaporator returns to the **Run Select** screen.

Drain: The system opens the drain valve and pumps any waste solvent into the Waste Solvent Container.

Defrost: Enter the defrost menu. Select **START** to begin the defrost cycle.



The evaporator defrosts the condenser until solvent defrost is automatically detected. Alternatively, press the **STOP** key to end the defrost cycle.

Shutting Down

Enter the **Select Run** screen and press the **STOP** key.

SELECT RUN						
No.	Name	Solvent	Vol.	Holder	T.	Nxt
91	Example	EtAc+Hex	1ml	Deep We	40	0
92						
93	PURIFICATION	WATER\ACETONITRILE	10ml	18 X 15	40	0
94	SOLID PHASE SYNTH	TFM\DCM	2ml	DEEP WE	40	0
95	COMPOUND			EEP WE	40	0
96	OLIGO SYN			EEP WE	55	0
97	SOLUTION			4 X 15	40	0
98	SOLUTION PHASE 2	DCM\METHANOL	1ml	DEEP WE	40	0
99	MAINTENANCE TEST 1		0ml		35	100
100	MAINTENANCE TEST 2		0ml		35	0
GENEVAC LTD						
MENU						
START EDIT GRAPH CLEAR COPY TO DRAIN DEFROST FLUSH OPTIONS						
HELP						
Select Run with ↑↓ , Menu Item with ↔ keys, press START to execute Starts selected Run using parameters stored						

Select **Y** to shut down the evaporator.

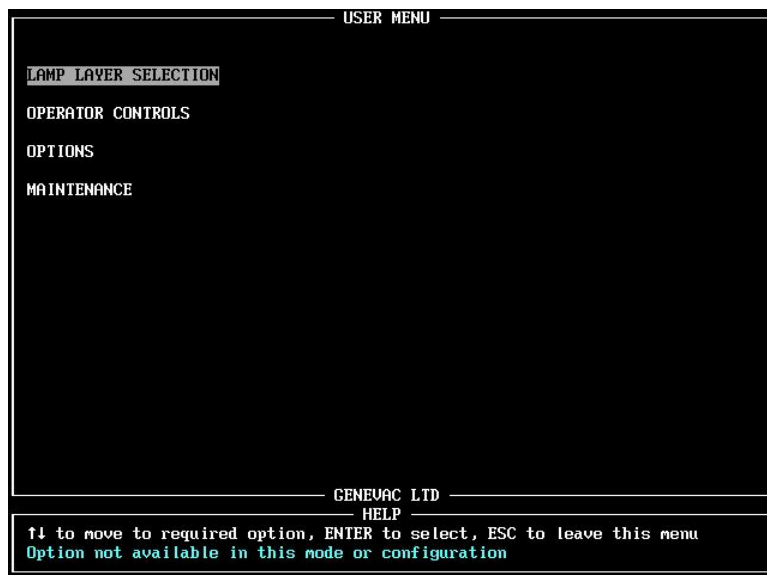


The evaporator continues to purge the vacuum pump for a time period indicated on the screen. Press the **START** key at any time to restart the evaporator.

Do not turn off the evaporator until the **Purge Time** has elapsed.

Options Menu

Select Options on the horizontal Menu bar.



Lamp Layer Selection: Disable associated lamp layers if 2nd or 3rd rotor levels are not used.

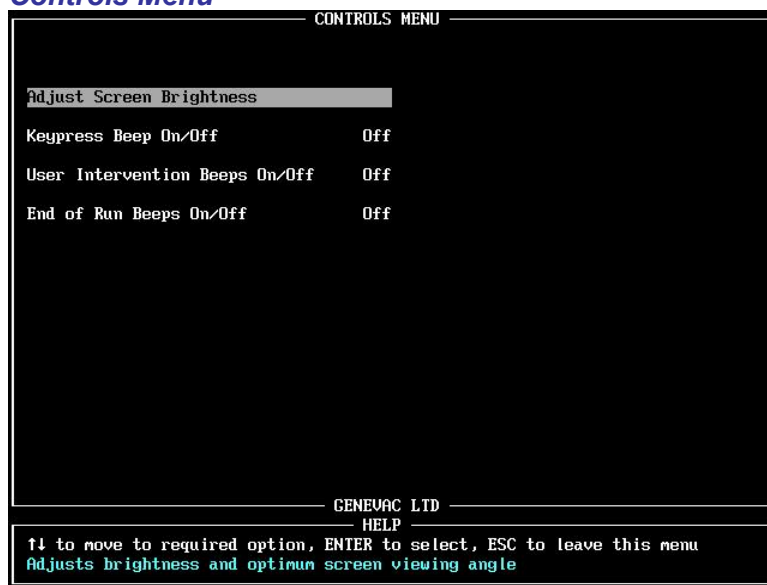
Operator Controls: Opens the **Controls Menu**.

Options: Enable **Auto Shutdown** and **Pre-Run Checklist**.

Maintenance: For engineer use only.

Status: Displays information relevant to fault diagnostics.

Controls Menu



Adjust Screen Brightness: Adjusts the screen viewing angle. The function is not available on newer instruments fitted with high contrast screens.

Keypress Beep On / Off: Turns off the key-press beep response.

User Intervention Beeps On / Off: Turns off the user-intervention beep response.

End of Run Beeps On / Off: Turns off repeating end of run alarm.

Press the **ESC** key to leave these menu screens.

Starting a Run

This section describes the steps required to carry out a typical evaporation run for DMSO. In this example, two sets of eight 28 x 60 mm scintillation vials, each containing 5 ml of DMSO are placed in solid aluminium sample holders.

Switch on the HT8 or HT12-SII evaporation system, select the **Run Data** screen, go to an unused run location and input the following data:

Run Data Locked	No
Next Run Number	0
Run Name	DMSO 28 x 60 mm tubes
Sample Holder Type	Solid Aluminium 28 x 60
Sample Volume	5
Solvent	DMSO
SampleGuard Control Temperature	40
SampleGuard Control Channel	1
CoolHeat Enable Pressure	100
Chamber Temperature Control	40
Rotor Speed	Low
Heat Off Elapsed Time	01:40
End of Run Elapsed Time	01:40
Controlled Pressure	Full Vacuum

Once the data is entered, the run profile is stored for future use.

The **SampleGuard** control temperature is set to 40°C and the chamber pressure to full vacuum. The chamber is preheated to 40°C to prevent solvent condensing in the chamber.



**SampleGuard Probe
Location Hole**

Dispense the DMSO samples into the vials and place the sample holders into the sample swings.

Load the sample holders and swings into the evaporator, ensuring that the vials, sample holders and sample swings are correctly and securely located. Refer to the section entitled: **Loading the Rotor** for details.

Position the channel 1 SampleGuard probe in its locating hole in the sample holder.

Position the channel 2 sample SampleGuard probe in an outer tube or well. Refer to the section entitled: **Using SampleGuard** for details.

Always ensure that the tip of the sample probe is located at the bottom of the tube.

Close the evaporator door and press the **START** key.

The display indicates that the chamber is warming up, this takes approximately twenty minutes. The system starts automatically when the chamber temperature reaches the **Chamber Temperature Control** value.

Some time can be gained by preheating solid aluminium sample holders to 40° C in an oven.

Other evaporation run profiles are suggested in the section entitled **Optimising a Run**.

The Evaporation Process

This section examines the process that occurs within the evaporator during the run. When the previously described evaporation run starts, the pressure drops steadily as air is evacuated from the chamber and condenser. The CoolHeat lamps operate when the pressure reaches 100 mbar and, because the chamber is pre-heated to 40° C, the sample holders quickly reach the pre-set temperature. The **SampleGuard** maintains the 40°C temperature ceiling.

As solvent starts to evaporate, the rate of pressure change reduces. The temperature of the sample drops due to evaporative cooling. Provided there is sufficient vacuum, the drop in sample temperature occurs even though the CoolHeat lamps are on, i.e. all the energy goes into evaporation and not into sample warming.

The evaporation rate and vapour flow drops until the pressure stabilises and a steady state is reached. There is then normally a long period of constant pressure and vapour flow whilst the bulk of solvent is evaporated.

As the solvent approaches dryness, the evaporation rate often reduces, causing the vapour pressure to drop. This is particularly noticeable with *oily* samples whose vapour pressure is very low. At this point, the sample temperature starts to rise, reaching the **SampleGuard** control temperature in a reasonably short time.

Optimising a Run

This section describes the general principles by which the Series II control software can optimise evaporation rates.

Limiting the Chamber Pressure

Pressure control can be used to significantly increase the evaporation rate for aqueous mixtures of greater than 30% water when contained in glass tubes and beakers. When evaporating these mixtures at pressures of less than 4 mbar, ice forms, extending the evaporation time. By controlling the evaporator at a pressure of 6 mbar, ice formation is prevented and the time required for evaporation can be reduced by up to 30%.

Linking Runs

Mixtures of water and methanol in deep well microtitre plates however, respond entirely differently. At full vacuum, a reasonable evaporation rate is achieved. At 8 mbar, the time required for evaporation increases significantly.

When evaporating 40 ml fractions in 24 x 150 mm glass tubes, typical evaporation times for water / acetonitrile mixtures can be in excess of 8 hours. The same applies to larger volumes in beakers.

A reduced evaporation time can be achieved by setting the **SampleGuard** control temperature to 60°C for a defined period. Provided this period is not excessive, the sample temperature is likely to remain below 40°C, as result of evaporative cooling. This is the case even though the **CoolHeat** lamps remain on.

After this period, a second or a number of successive runs can be linked at reduced SampleGuard temperatures and different pressures. Some experimentation is necessary to ensure that safe sample temperatures are not exceeded.

Pre-programmed Runs

HT8 and HT12-SII evaporators, are pre-programmed with a number of evaporation methods. These *runs* are located at 51 to 60 of the **RUN** selection screen. Desired runs can be copied to other locations using the **copy to** function. Runs 61 through to 98 are all associated with these pre-programmed methods, and runs 99 and 100 are for use by Genevac service engineers, please do not alter them.

The pre-programmed runs are designed to cover a group of solvents, such as *high boiling point*. The following table provides a guide to the correct run selection. Each run is programmed to be used with a particular group of solvents. Therefore, it may be possible to program a more efficient method for just one of those solvents. For advice on programming a suitable method for a specific solvent or solvent mixture, please contact your Genevac representative, or go to applications@genevac.com.

The pre-programmed runs are designed to make use of all the appropriate features of the system and to provide optimal evaporation conditions. All methods make use of the automatic end of run detection feature, so that run times do not need be set, the system stops when all the samples are dry. Each run has a venting procedure linked to the end of the evaporation process. This helps to remove residual vapours from the chamber.



The preset programs will only function correctly if SampleGuard probe #1 is placed in the aluminium sample holder block or fast stack swing. See the section entitled: **Using SampleGuard** for details.

Run Number	Name	BP Range	Solvent Examples	Application
51	Volatiles - not DCM	60-90°C	Ethyl Acetate or Methanol	Single volatile solvents with no risk of bumping
52	Very Volatile & DCM	40-90°C	DCM (Methylene Chloride) or Chloroform	Volatile solvent mixtures, and DCM (Methylene chloride), where risk of bumping is high
53	Medium BP Solvents	90-155°C	DMF or Toluene	Medium BP solvent or mixture of medium BP solvents
54	Med BP & Volatile	40-155°C	DMF & DCM (Methylene Chloride)	Mixtures of medium and volatile solvents with high risk of bumping
55	Pre-heat Chamber for High BP solvents	-	No solvents should be present	Rapid pre-heat of chamber before a high BP solvent run, swings and samples must be removed before this method is used.
56	High BP 140+	140-190°C	DMF, DMAc, DMSO	High BP solvents
57	Very High BP Solvents	190°C +	NMP, DMI	Very high BP solvents
58	HPLC Fractions	-	Water & Acetonitrile or Methanol	HPLC fractions
59	Aqueous only	100°C	Water	Water only
60	Remove stubborn solvents	-	Any	Drying samples, which have not been fully dried using another method. The condenser must be empty before this method is used

Getting the Best from the System

Routine Checks

For high boiling point solvents such as DMSO, NMP, DMF and DMI the best evaporation rates are achieved at pressures better than 0.5 mbar. Carry out the following checks regularly to ensure optimum vacuum performance.

- Check security of all clamped joints.
- Drain the condenser pots before every run.
- Check the pump exhaust catch pot regularly and drain as necessary.
- Keep a log of the time taken for your system to reach full vacuum and use it to indicate deterioration of performance due to seal ageing and wear.

Tips for Improving System Performance

- Increase evaporation speed for high boiling point solvents by pre heating the aluminium sample holders.
- Use the link run facility when evaporating solvent mixtures with large differences in boiling point. See the section entitled: **Optimising a Run** for details.

Further Checks for CVP Pumps

- Check the pump oil level weekly.
- Ensure that the oil level is **within** the notch on the dipstick.



- Never apply vacuum to a heated condenser (after defrosting) containing traces of liquid DMF, DMSO or NMP. This causes pump contamination.

Run Parameters Quick Reference

Run Data Locked

Set to **Yes** to protect the run data from inadvertent changing.
Press the **ENTER** key to toggle between **Yes** and **No**, lock the data after completing the run entry.

Next Run Number

Enables the creation of multistage runs by linking runs in any order. Enter the number of the run that is required to follow the current run. Runs may also be *looped* by linking to themselves, this makes the run cycle repeatedly until interrupted by a press of the **STOP** key.

Run Name

Identifies the run profile.

Sample Holder Type

Select the sample holder type from the drop down menu or select **OTHER** and enter the holder name.

Sample Volume

Enter the volume (of the individual samples) in mls.

Solvent

Select the solvent from the drop down menu or select **OTHER** and enter the solvent name (no more than 31 characters).

Assuming that recognised data was entered in the preceding fields, the **Auto Program** option appears. Select this to automatically programme the remaining run parameters. Auto programmed parameters appear highlighted in **cyan** and may be overwritten manually.

SampleGuard Control Temperature

Enter the maximum temperature to which the samples holders are heated during evaporation. The system's ability to control the sample holders at lower range temperatures is dependant upon the solvent boiling point (i.e. Evaporative cooling) and the ambient temperature within the vacuum chamber.

This can be set within the range: -20 to +70° C.

The default setting is 30° C if no other value is entered.

Note: this is not available for **very low** rotor speed.

Sample Guard Control Channel

Channel 1 is assigned as the control channel by default (channel 1 probe is placed in the sample holder, channel 2 probe is optionally placed in the sample). Assign control to channel 2 and place the channel 2 probe in the sample holder, only if a fault affects channel 1 or its associated probe.

CoolHeat Enable Pressure

Enter the pressure below which the lamps turn on.

This can be in the range of 5-400 mbar.

The default setting is 400mbar if no other value is entered.

Chamber Temperature Control

Select from 3 available options:

Wait for Chamber to Heat: Range 0°C to 45°C.

Wait for Chamber to Cool: Range 0°C to 45°C.

Automatic Control: The control temperature is automatically set to an optimised figure for the recognised solvent / mixture selected. Enter the chamber temperature required before spin-up commences.

Note: do not set the **Wait for Chamber to Cool value** below the ambient temperature.

Rotor Speed

Select from 3 available options:

High: Maximum acceleration applied to samples – 500G

Low: Maximum acceleration applied to samples – 300G

Very Low: Maximum acceleration applied to samples – 50G

The default setting is **Low** if no other selection is made. If the **Dri-Pure** option (described later) is selected, it over-rides the rotor speed setting and sets **High** speed.

Heat-Off Option

Select from 3 available options:

- **Elapsed Time:** This can be in a range of 0 (lamps off for the entire run) to 99 hours 59 minutes.
- **Heat Flow:** Automatically detects end of run by monitoring evaporative heat demand.

Heat Flow can operate with 1 SampleGuard probe fitted only. The control channel probe **must** be placed in the sample holder. The system detects dryness by monitoring the demand for heat input (proportional to the rate at which the solvent is evaporating).

Heat flow may not work at very low control temperatures or with very low sample loading. In these instances the proportion of evaporation energy drawn from ambient temperature becomes significant enough to affect the system's ability to detect heat input.

For applications where **Sample Guard** control temperature or low sample loading is low, use either **Elapsed Time** or **Sample Detection** methods for the **Heat-Off Option**.

- **Sample Detection:** Automatically detects end of run by monitoring sample cooling in liquid phase.

Requires both SampleGuard probes to be fitted, Channel 1 monitors the swing / holder temperature. Channel 2 is placed in a central well of the sample holder and monitor the sample temperature.

The system monitors the difference between the control temperature and the sample temperature. As vacuum is applied the sample boiling point drops and the samples cool whilst in liquid phase, their heat energy being absorbed by evaporation (evaporative cooling). The system detects dryness as the temperature of the dried product rises.

End-of-Run Option

Set the run time duration.

End-of-Run Elapsed Time

Select the elapsed time after which the run is stopped.
This can be in the range of 1 minute to 99 hours 59 minutes.

End of Run Defrost and Drain

Select **Yes** or **No**. Only available on LyoSpeed or automated systems. Typically used to remove mixed solvents.

Pressure Control Regime

Select from the following options:

- **Full Vacuum:** Reduces the chamber pressure to the maximum vacuum capability of the pump.
- **Controlled Pressure:** Reduces the chamber pressure to a set value. Enter a value for the required **Controlled Pressure** between 0 and 500 mbar.
- **Dri-Pure:** Reduces the pressure to 50 mbar by equal increments over a 40 minute time period. Then goes to the control pressure. This *ramp* feature is particularly useful for preventing bumping*.
- **Variable Dri-Pure:** As **Dri-Pure** but with additional user settable parameters for the *ramp* start pressure, the *ramp* end pressure, the *ramp* duration.

* Bumping: the violent and uncontrolled boiling of solvents, resulting in cross contamination of samples.

Note: The **Coolheat** function is disabled during the **Dri-Pure** cycle.

Problem Prevention

Condensation

Solvent condensation within the evaporator occurs when the chamber walls are cooler than the solvent vapour. This is most likely to occur with high boiling point solvents such as NMP, DMI, DMSO and possibly DMF.

Pre-heat the evaporator chamber to prevent this happening. To do this, enter the **Run Data** screen, set the Minimum Chamber Temperature field to 40°C and start the run. The chamber takes approximately 20 minutes to reach this temperature. Note the CoolHeat lamps do not operate during this pre-heating cycle.

If condensation occurs unexpectedly, switch off the lamps by entering 0 into the Heat off Elapsed Time field on the **Run Data** screen.

Do not pre-heat the chamber when evaporating volatile solvents such as TFA, acetonitrile or methanol.

TFA Creep

TFA exhibits the property of *creeping*. This is the movement of the TFA in liquid phase up the inside of the tubes, vials or microtitre plates. Problems can arise if solvent containing product is deposited, in this way, on the top face of plates. Solvent and product may also be thrown onto the side of the chamber and the Quartz glass lamp windows as the rotor spins.



Whilst the solvent evaporates, the product becomes carbonised by the heat from the lamps and forms sites where crack propagation can occur. Inspect the lamp glass at regular intervals and clean with a lint free cloth and acetone.

Contact Genevac Service if the contamination becomes excessive and carbonised as shown.

A range of specialised sample holders is available to protect the Quartz lenses. Contact Genevac Sales for details.

Bumping

Bumping can also cause product to deposit on the glass lenses as previously described. It is unpredictable and may occur with any solvent or mixture, it is also a potential source of cross contamination between samples.

Avoid bumping by selecting the **Dri-Pure** option from the **Pressure Control Regime** drop down menu on the **Run Edit** screen. This option sets a high rotor speed and avoids super-heating the samples by reducing the pressure in the vacuum chamber by gradual increments. Evaporation begins progressively and samples are contained due to the higher centrifugal acceleration.

Maintenance

Refer to the section entitled: **Getting the Best from Your System** for details of routine maintenance checks. This section describes some further proactive maintenance procedures.



Excessive build up of debris on the pivoting faces of the sample swings and rotor can cause the sample swings to stick in the out position when the rotor stops. This can cause unrecoverable sample loss.

Cleaning the Sample Holders and Swings

Regular inspection and maintenance of swings and sample holders should be performed at least monthly. The following inspection routine is mandatory following any tube breakage or solvent spillage. Never use wet swings or holders in an evaporator.

Visually inspect the sample holders monthly. Clean off any debris, especially in the sample holder wells as this may cause high points that lead to stress in the glassware, resulting in breakage. The main cause of repeat glassware breakage is fragments from a previously broken tube. Solvent can stick the glass fragments to the holder making it difficult to remove. Residual solvent or sample material must be cleaned off.

Superficial surface damage (e.g. scratches) do not affect the performance of a holder or swing. However, if there is any structural damage (if any part of the holder, swing or rotor is bent or deformed) do not use it. Contact Genevac Service for evaluation.

Cleaning the Chamber, Rotor and Swings

TFA creep can cause debris deposits on the inside of the chamber and on the Quartz glass, see **Problem Prevention** for details. Routinely inspect the inside of the chamber and quartz glass for build up of debris and potential contaminants. Check the sample swings are able to move freely.

Clean the Quartz Glass using a suitable solvent (such as methanol or acetone) and a lint free cloth or paper towel. Clean the swings and rotor in the same manner. Take care to avoid solvent contact with the outside paintwork and accessories of the chamber.

Scroll Pump

Check the pump exhaust catch pot regularly and empty it as necessary. No further routine maintenance is required.

Further Notes for CVP Pumps

Check the pump oil level weekly. Ensure the oil level is **within** the notch on the dipstick. Top it up if necessary using the correct grade of silicon oil.



Do not apply vacuum to a recently defrosted condenser containing traces of liquid DMF, DMSO or NMP. This is likely to cause pump oil contamination.

Correct Oil level – centre of dipstick notch

<i>Pump Type</i>	<i>Correct oil</i>	<i>Part Number</i>
CVP100/CP	SCF025	AS1616 (1 litre)
CVP100/CPC	Crylin 614	AC2310 (1kg)

The pump must have an adequate supply of cooling air and a minimum 300mm of free space adjacent to the heat exhaust. Do not place the pump in a space with restricted airflow, such as a cupboard. Planned

Maintenance and Service

Whilst every effort is made to design and manufacture the HT4-SII and HT4X-SII evaporation systems to the highest build quality and to provide assured reliability, there will be some degree of wear and ageing of the seals and bearings of the chamber, condenser and pump. The extent of wear and ageing depends on the utilisation of the system, the severity of temperature cycling and the nature of the solvents used.

Some solvents may eventually cause pin-hole corrosion in the connecting tubes, resulting in a decline in performance. A gradual decline in system performance may not be noticed if it occurs over a period of time. Monitor the system performance by keeping a weekly log of the time taken for the system to reach full vacuum and the full vacuum achieved.

In order to maintain peak performance and avoid costly and unscheduled down time, Genevac strongly recommend implementing a schedule of planned maintenance. Changing parts in the field is complex and demands a high level of skill. To this end, Genevac offers a range of preventative maintenance, service and breakdown contracts.

Moving the System

Refer to the section entitled: **Positioning the Evaporator**. Refer also to the following notes if it is necessary to move a system to a new location. Address the following key points before moving an HT4_SII or HT4X-SII evaporation system.

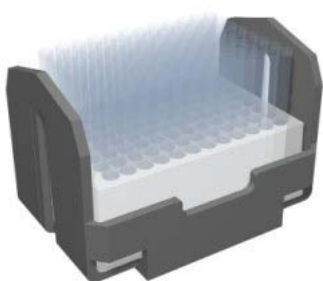
- Clear sufficient bench or fume cupboard space to accommodate the system at the new location.
- Ensure there is access to a power supply (two separate mains sockets are required).
- Ensure there is adequate ventilation.
- Make provision for the drainage of waste solvents.
- Make provision for the extraction of the pump exhaust.
- Defrost and drain (and if possible, flush) the condenser pots.
- Remove the sample swings from the rotor.
- Remove and retain all the clamps, seals, tubes, cables and instructions.

Additional Equipment

Genevac supply a range of swings and sample holders which may be utilised to adapt existing systems for new applications. All sample holders are of a solid aluminium construction and are black anodised to improve heat absorption. Solid aluminium tube holders provide even heat distribution for uneven drying loads. Maximum contact areas provide good physical support, optimum heat transfer and even heat distribution

The following accessories are an example of the many ways in which Genevac can assist in developing your system for the future.

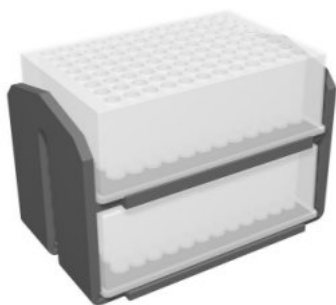
For further information, or to discuss any requirements, please call Genevac Sales using the contact details on the back cover of this User Manual.



Side Bridge



FastStack for shallow well plates



FastStack for deep well plates



Flask holder



One piece holder for 50ml Tubes



Sample Genie

Auto Defrost Drain

Auto-Defrost and Drain is a condenser option for the Genevac HT4X, HT8 and HT12 series 2 evaporation systems, and is standard on the HT-24 Workstation. Auto-Defrost and Drain enables the system to:

- Automatically drain the condenser of volatile solvent(s) between method stages.
- Automatically drain the condenser at the end of the method.

The Auto-Defrost and Drain system requires no user intervention. When performing an intermediate drain (i.e. mid method) the system also performs a short defrost in case residual solvent from a previous use is frozen in the outlet pipe of the condenser. The intermediate drain removes volatile solvents which do not freeze at temperatures above -50°C .

Benefits of Auto-Defrost & Drain

The principal benefit of the Auto-Defrost and Drain function is to eliminate volatile solvent(s) from the condenser. The volatile component of a mixture boils off first and is collected in the condenser. To remove the higher boiling point solvents, low pressures must be applied later in the process. However, reducing the pressure causes the previously condensed volatile solvent to re-boil in the condenser, generating a very large volume of vapour which must exit through the pump. Until all this vapour is pumped away (which can take several hours), the system cannot achieve a lower pressure. The volatile solvents are said to *spoil* the vacuum, and so the higher boiling point solvents do not boil. Vacuum spoiling affects final dryness of samples, or in the very worst cases, the ability to evaporate higher boiling point solvents at all. To overcome this problem, manual intervention was previously required to drain the condenser after the volatile solvent was captured in the condenser. This draining operation can now be automated. An additional benefit of Auto-Defrost and Drain is that volatile solvents are collected and can be disposed of safely, reducing VOC emissions.

Evaporators equipped with the Auto-Defrost and Drain facility are identified by a **Lyo Speed Enabled** label:



The Auto-Defrost and Drain option also appears in the run menu when programming methods.

Flexibility

A Genevac evaporator enhanced with Auto-defrost and Drain functionality can be used as part of the **LyoSpeed™** process. In addition it can also deliver improved results when working with any mixture of solvents with differing boiling points.

Auto-defrost and drain helps achieve excellent final dryness when evaporating HPLC fractions. It also facilitates automated drying of DCM and DMSO or DMF mixtures. These are otherwise almost impossible to evaporate without draining the DCM before tackling the higher boiling point solvent.

How it Works

The Auto-Defrost and Drain condenser includes the following features:

- Enhanced condenser draining with minimum hold-up
- Automation of the drain valves
- Specific defrost of condenser outlet pipe
- Mid-method short defrost and drain to remove volatiles
- Automated full defrost and drain at end of method

Auto-Defrost and Drain appears as an option in the **Run Data** screen when entering the method parameters. The evaporator performs in one of two ways depending on whether the method is part of a linked series of methods:

- If the method stands alone and is not linked to any other method, the system performs a full defrost at the end of the method, and then drains the condenser automatically.
- If the method is linked (other methods follow it) the system performs a short defrost, sufficient only to defrost any frozen solvent trapped in the outlet pipe, then drains the condenser. When this is complete, the following linked method continues automatically.
- If the method has other methods linked to it, but is the last in the chain, then at the end of the method the system a full defrost and drains the condenser automatically.

When to use Auto-Defrost and Drain

Auto-Defrost and Drain has two main functions:

1. To automatically drain the condenser mid-process to eliminate volatile solvents, enabling the system to reach full vacuum in the later stages of evaporation. This allows the system to achieve better final drying of the sample, and to get better evaporation of mixtures of solvents with widely differing boiling points.
2. To automatically defrost and drain the condenser at the end of the method, saving the user time. This means that for an overnight method which finishes before the user comes to the Lab in the morning, the system is defrosted, drained and is ready to use straight away. This has a benefit for any solvent.

The second of these requires no further explanation, however, there are a number of applications for the first; the intermediate defrost and drain. These include:

- Drying of HPLC fractions where high vacuum in the final stages of evaporation is beneficial.
- Use of the Genevac fast lyophilisation method for HPLC fractions, where high vacuum in the latter stages of the process is essential.
- Evaporation of DMF or DMSO mixed with a volatile solvent, e.g. dichloromethane (also known as DCM or methylene chloride) where the mixture of solvents necessitates high vacuum for the high boiling point solvent which is removed last, but where the volatile solvents removed first, sit in the condenser and spoil the level of vacuum achieved in the latter stages of the run.

When not to use Auto-Defrost and Drain

There are certain circumstances when Auto-Defrost and Drain should not be used. This is to protect the system and / or samples from solvent damage. Do not use Auto-Defrost and Drain in the following circumstances:

- Evaporating TFA: intermediate (short) defrost and drain is permitted. Full defrost and drain at the end of the method should not be performed if there is residual TFA in the condenser. Perform a manual defrost for 10 minutes, manually stop the defrost cycle, and drain the system.
- At the end of a lyophilisation method: water vapour may travel back from the cold trap to the chamber and cause the powders to collapse. Remove the samples first, then defrost and drain.

Drying HPLC Fractions

The best optimised evaporation method has the following stages:

1. First Stage - removal of the organic phase

- a. Use Variable Dri-Pure™ to prevent bumping:
Ramp the pressure down from 175mbar to 40 mbar (acetonitrile) or 45mbar (methanol) in 20 minutes at high rotor speed (500g). Control the pressure at 40mbar for Acetonitrile and 45mbar for Methanol, these control pressures are chosen to keep the boiling point above 0°C (i.e. prevent the water freezing).
- b. Auto-Defrost and Drain at the end of the stage, this is a short defrost (in case a small amount of water is frozen in the drain port). The system then drains the organic solvent from the condenser, allowing high vacuum levels to be achieved later in the run.
- c. Consult your local Genevac representative for advice on the evaporation time for this stage. However, a good rule of thumb is that for 50 / 50 Acetonitrile water the overall length of this stage should be approximately 1/3 as long as the length of stage 2 (which the system determines automatically).

2. Second Stage - removal of the aqueous phase.

- a. Control the pressure at 8mbar (the BP stays above 0°C and the water cannot freeze).
- b. Select high rotor speed (promoting good convection & hence heat flow in the solution).
- c. End the method using **Heat Flow** rather than specifying the duration for the stage.

3. Third Stage - drying the stubborn samples.

- a. Run the system at Full Vacuum for 1 to 3 hours.
- b. Experimentation is required to optimise this stage. Different compounds require different times to reach final dryness, highly polar molecules often take the longest.
- c. Auto-Defrost and Drain at the end of the stage - this is a full defrost and drain, as there are no more stages to follow.

See appendix A for programming details.

Please contact your local Genevac representative for further details on run times, alternatively please e-mail Applications@Genevac.com

Lyophilising HPLC Fractions

The best optimised evaporation method has the following stages:

1. Follow stage 1 of the **Drying HPLC Fractions** method to remove the organic solvent.
2. Cool the samples, concentrating some of the water without any heat input. Set the controlled pressure to 8mbar, to boil away some of the water without any heat input, this cools the holders and helps achieve good freezing in the next stage. It may be possible to concentrate more water at this stage, for very large or water heavy fractions please contact Genevac for advice.
3. Freeze all the samples using high vacuum. Run the system at Full vacuum with no heat for 1 hour
4. Lyophilise the samples to dryness, using heat if possible to speed up the process. Run the system at full vacuum, trial and error will determine the length of time that Lyophilisation takes

Please contact your local Genevac representative for further details on run times, alternatively please e-mail Applications@Genevac.com

Notes on Lyophilisation

- Some experimentation is required to set up a new fast lyophilisation method. Several trials are required to optimise the method and produce a fully functioning process. Genevac are able to provide remote assistance by analysing evaporation data collected using the **Genevac Data Logging Software**. An adjacent laptop or PC is required to collect this data.
- Use both sample guard probes, place probe 1 in the sample holder to control the temperature, place probe 2 in a central sample to monitor the process. This is essential during method development. It indicates when the sample freezes and when the lyophilisation process ends.
- Fill all positions of the sample holders when performing lyophilisation, samples in partially filled holders may not freeze.
- Do not use this method to attempt lyophilisation of samples that are grossly insoluble in water. These samples crash out once the organic solvent is removed from the mixture.
- Never use Auto-Defrost and Drain on the final stages of a lyophilisation method – solvent vapour from the condenser may re-enter the samples and cause the powder to collapse. Feedback from some Genevac users suggests that this is worse when samples are TFA salts, whereas HCl salts are reportedly more robust. These observations have not been proven.
- Remove the samples from the system before defrosting the condenser.

Drying Mixed High and Low Boiling Point Samples

The actual method required depends on the solvents in the mixture. A mixture of DMF or DMSO and DCM is outlined in this illustration. For other details of mixtures, please contact Genevac.

1. Select **Variable Dri-Pure** to prevent bumping. Ramp from 750mbar to 70 mbar in 55 minutes.
2. Evaporate the DCM at 65mbar, and collect it all in the cold trap.
3. Select **Auto-Defrost and Drain** – this is a short defrost and drain to remove the organic solvent from the condenser, allowing high vacuum levels to be achieved later when drying the high boiling point solvent.
4. Evaporate the high boiling point solvent. Select **full vacuum** – end the method using **heat flow**.
5. Dry any stubborn leftover solvent. Select **full vacuum** and let it run for 1 hour.
6. Select **Auto-Defrost and Drain**, this is a full defrost and drain.

See appendix C for programming details.

Please contact your local Genevac representative for further details on run times, alternatively please e-mail Applications@Genevac.com

Programming Guides

The programming guides on the following pages show examples of run profiles for some common applications.

Drying HPLC Fractions

	Drying HPLC Fractions		
Run number	1	2	3
Run Data Locked	N	N	N
Next Run Number	2	3	0
Run Name	Fractions	Fractions part 2	Fractions part 3
Sample Holder Type	Your Holder	Your Holder	Your Holder
Sample Volume ml	Your Volume	Your Volume	Your Volume
Solvent	MeCN or MeOH & H ₂ O	MeCN or MeOH & H ₂ O	MeCN or MeOH & H ₂ O
SampleGuard Control Temp C	40	40	40
SampleGuard Control Channel	1	1	1
CoolHeat Enable Pressure	50	50	50
Chamber Temperature control	preheat to 25	preheat to 30	preheat to 30
Rotor Speed	High	High	Low
Heat-Off Option	Elapsed Time	Heat Flow	Elapsed Time
Heat-Off Elapsed Time	<i>ask for time</i>	-	01:00 - 03:00
End-of-Run Option	Elapsed Time	-	Elapsed Time
End-of-Run Elapsed Time	<i>ask for time</i>	-	01:00 - 03:00
Auto Defrost & Drain	Y	N	Y
Pressure Control Regime	Variable Dri-Pure	Controlled Pressure	Full Vacuum
Dri-Pure Start Pressure (mbar)	175	-	-
Dri-Pure End Pressure (mbar)	40 for MeCN 45 for MeOH	-	-
Dri-Pure Ramp Time	20	-	-
Final Control Pressure	40 for MeCN 45 for MeOH	8	-

Notes

Remove organic phase

Remove most of the aqueous phase

Remove final "stubborn" solvents

Fast Lyophilisation of HPLC Fractions

	Lyophilising HPLC Fractions			
Run number	1	2	3	4
Run Data Locked	N	N	N	N
Next Run Number	2	3	4	0
Run Name	Fractions Lyo	Fractions Lyo part 2	Fractions Lyo part 3	Fractions Lyo part 4
Sample Holder Type	Your Holder	Your Holder	Your Holder	Your Holder
Sample Volume ml	Your Volume	Your Volume	Your Volume	Your Volume
Solvent	MeCN or MeOH & H ₂ O	MeCN or MeOH & H ₂ O	MeCN or MeOH & H ₂ O	MeCN or MeOH & H ₂ O
SampleGuard Control Temp C	40	0	0	40
SampleGuard Control Channel	1	1	1	1
CoolHeat Enable Pressure	50	50	50	50
Chamber Temperature control	preheat to 25	preheat to 0	preheat to 0	preheat to 30
Rotor Speed	High	High	Low	Low
Heat-Off Option	Elapsed Time	Elapsed Time	Elapsed Time	Elapsed Time
Heat-Off Elapsed Time	<i>ask for time</i>	0:00	00:00	05:00
End-of-Run Option	Elapsed Time	Elapsed Time	Elapsed Time	Elapsed Time
End-of-Run Elapsed Time	<i>ask for time</i>	0:30	01:00	10:00
Auto Defrost & Drain	Y	N	N	N
Pressure Control Regime	Variable Dri-Pure	Controlled Pressure	Full Vacuum	Full Vacuum
Dri-Pure Start Pressure (mbar)	175	-	-	-
Dri-Pure End Pressure (mbar)	40 for MeCN 45 for MeOH	-	-	-
Dri-Pure Ramp Time	20	-	-	-
Final Control Pressure	40 for MeCN 45 for MeOH	8	-	-

Notes

Remove MeCN

Cool the water

Freeze the water

Lyophilise the water

Mixtures of High and Low Boiling Point Solvents

	Drying High - Low Mixtures		
Run number	1	2	3
Run Data Locked	N	N	N
Next Run Number	2	3	0
Run Name	High - Low Mix	High - Low Mix part 2	High - Low Mix part 3
Sample Holder Type	Your Holder	Your Holder	Your Holder
Sample Volume ml	Your Volume	Your Volume	Your Volume
Solvent	DMSO or DMF & DCM	DMSO or DMF & DCM	DMSO or DMF & DCM
SampleGuard Control Temp C	40	40	40
SampleGuard Control Channel	1	1	1
CoolHeat Enable Pressure	50	50	50
Chamber Temperature control	preheat to 15	preheat to 40	preheat to 30
Rotor Speed	High	High	Low
Heat-Off Option	Elapsed Time	Heat Flow	Elapsed Time
Heat-Off Elapsed Time	ask for time	-	00:00
End-of-Run Option	Elapsed Time	-	Elapsed Time
End-of-Run Elapsed Time	ask for time	-	01:00
Auto Defrost & Drain	Y	N	Y
Pressure Control Regime	Variable Dri-Pure	Full Vacuum	Full Vacuum
Dri-Pure Start Pressure (mbar)	750	-	-
Dri-Pure End Pressure (mbar)	70	-	-
Dri-Pure Ramp Time	55	-	-
Final Control Pressure	65	-	-

Notes

Remove MeCN

Dry high BP solvent

Final Drying

Technical Data

Mechanical data		Condenser	
Max rotor speed	1300 standard 1800 Dri-Pure	Temperature max low	-45°C (HT4 SII) -50°C (HT4X SII)
Max force	300-500G	Temperature max high	+60°C
Drive system	Direct	Vacuum pot capacity	1.5 litres (HT4 SII) 2.3 litres (HT4X SII)
Operational imbalance	85g	Exhaust pot capacity	0.4 litres
Max load	4x1.5kg @ 500G	Condenser level	No
IR lamps number	2	detector	316 Stainless steel
Weight	109 kg (HT4 SII) 118 kg (HT4X SII)	Condenser chamber	Stainless steel / PTFE
Vacuum System		Vacuum Pump - Scroll	
Pressure resolution	1-1000 mbar	Weight	28kg
Vacuum control	1-1000 mbar	Maximum vacuum	0.15 mbar (50Hz) 0.12 mabr (60Hz)
Dri-Pure	Yes	Flow rate	3.6 m ³ h ⁻¹
Auto vacuum vent valve	Yes		
Rapid defrost	Yes		
Ultimate system vacuum	0.4 mbar		
Vacuum pump - CVP		Dimensions	
Weight	52 kg	Evaporator (W xD x H)	540 x 640 x 577mm (HT4) 590 x 640 x 590mm (HT4X)
Maximum vacuum	0.15 mbar	Scroll pump	530 x 305 x 398
Flow rate	3.6 m ³ h ⁻¹	CVP pump	540 x 290 x 405
Electrical			
Power Supply single phase 9A	230V, 50Hz 208V, 60Hz		

Operating Environment		Storage Environment	
Ambient temperature	15°C to 30 °C	Ambient temperature	-10°C to 60°C
Relative humidity	10 – 60 %	Relative humidity	10 – 80 %
Altitude	Sea level to 12,000m	Altitude	Sea level to 12,000m

EC Declaration of Conformity

We **Genevac Limited**, declare that this product: **Series II Evaporating System**, Complies with the relevant Essential Health and Safety Requirements of the European Machinery Directive (98/37/EEC), the EMC Directive 89/336/EEC, and the Low voltage Directive 73/23/EEC.

Conformity is demonstrated by compliance with the following specifications: -

EN 60204-1:2006	Safety of machinery– Electrical equipment of machines-Pt 1 General Requirements.
EN 249: 1992	Safety of machinery– Safety distances to prevent danger zones being reached by upper limbs.
EN 1088: 1995 + A1:2007	Safety of machinery. Interlocking devices associated with guards. Principles of design and selection.
BS EN ISO 12100 pts 1 & 2:2003	Safety of Machinery - Basic concepts, general principles for design.
BS EN 50082-1: 1998	Electromagnetic compatibility-Generic immunity standard.
BS EN 61010-1:2001	Safety requirements for electrical equipment for measurement, control and laboratory use, general requirements.

Warranty Statement

This product is guaranteed for period of 12 months from the date of delivery to site. In the unlikely event of any defect arising due to faulty materials or construction resulting in system failure, the unit will be repaired free of charge. This includes all labour and component costs incurred.

This warranty is subject to the following provisions:

- 1.** System must be sited, installed and operated in accordance with operator instruction manual.
- 2.** The system is only used for purpose it was sold, and in accordance with Genevac published compatible solvent list.
- 3.** The regular cleaning and preventative maintenance schedule must be adhered to as detailed in operator's manual.
- 4.** The warranty does not cover accidental damage, misuse, modifications or inappropriate repair by untrained personnel.
- 5.** The warranty does not cover the following consumable items: Sample Guard thermocouple probes, control fuses.

Failure to adhere to the above may result in the costs of repairs being charged.

Amendment Control Form

Issue	Reason for Change	Date Issued
2 – 7	Included in this issue.	
2-8	Integration of “The Importance of Safe Loading” Document. Introduction of new Pre-Set runs 51 -100 within the software. Introduction of Scroll Pump.	01 March 2004
2-9	Introduction of Warranty Statement	26 April 2004
2-10	Change to EU Declaration of Conformity	21 September 2004
2-11	Information on the correct use of SampleGuard Probes	28 September 2005
2-12	Introduction of Auto Defrost and Drain Option	21 August 2006
2-13	Introduction of Annex A – Getting the best from Auto Defrost and Drain	15 September 2006
2-14	Converted to print A5 Booklet	08 December 2006
2-15	Add Maintenance of Rotor, Swings and Sample Holders	09 April 2008
2-16	Update page footers	04 June 2008
3-1	Reformat layout, remove mega & HT12 references, default to current build version & include old builds as variances. Update Statement of Conformity.	5 December 2008
3-2	Update sample holder images.	26 January 2009

These instructions are correct at time of going to press and may be subject to change without notice.

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These operating instructions should be read before you use the Genevac HT4-SII or HT4X-SII evaporation system.

Your attention is drawn in particular to the section entitled: **Safety**



The evaporator should not be discarded in your regular disposal stream. Contact your Distributor or Genevac for proper disposal instructions.

Within the EU, it is Genevac's responsibility under the WEEE directive to provide for the recycling of their products.

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Useful Information

If you need to contact Genevac for assistance, use either the telephone or fax Hotlines shown.

It always helps Genevac Service if you have the serial numbers at hand for the components of your system

If you need to contact Genevac Sales for information on Service Contracts or products, use the telephone or fax Hotlines shown.

Alternatively, Email or visit our web site.

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Read these operating instructions before using the Genevac HT4-SII or HT4X-SII Evaporating Systems. Keep them near the system for easy reference. Your attention is drawn in particular to the section entitled: **Safety**.