Linkam Scientific Instruments

FDCS196

Freeze Drying Cryo Stage

USER GUIDE

Contents

Before Setting Up Your Equipment

Please register your products by going to www.linkam.co.uk and click on the product/software registration button.

You will need to register your equipment with us to:

- Activate your warranty and technical support
- Access the online setup videos
- Permanently unlock the Linksys32 software (if purchased)

If you have purchased Linksys32 software, please install the software first. This process will guide to register all of your products.

See Linksys32 manual for further installation instructions.

A CD with a setup videos is supplied with your system.

Important Notice

Please check that your Linkam equipment has not been damaged during transit. If there is any evidence of external damage DO NOT SWITCH ON ANY ELECTRICAL ITEMS.
Contact LINKAM SCIENTIFIC or their appointed distributor immediately. Your warranty may be impaired if Linkam is not informed of any transport damage within 7 working days of delivery.
NO attempt should be made to repair or modify the equipment in any way, as there are **no user replaceable parts**.
No attempt should be made to open the case except by qualified personnel as hazardous voltages are present.
In order to use this equipment successfully, please take time to read this manual all the way through before using it.

Warranty

This equipment has a warranty against defects in material and workmanship for a period of 12 months. Linkam will either repair or replace products that prove to be defective. For warranty service or repair, this product must be returned to Linkam or a designated service facility.
The warranty shall not apply to defects resulting from interfacing, unauthorized modification or misuse, operation outside of the environmental specifications for the product, or improper site preparation or maintenance.

Technical Support

Any technical questions or queries should be addressed to the Technical Support Department at the address shown on the back of this manual.

Equipment Maintenance

Use a small quantity of isopropyl alcohol with a soft cloth and gently wipe the surface. To clean the hotstage, use isopropyl alcohol (IPA) and cotton swabs. Take great care not to touch the platinum temperature sensor protruding from the side of the heating element. The sensor is very fragile.

Handling Liquid Nitrogen

To cool samples below room temperature a LNP95 liquid nitrogen pump is required. Please refer to your health and safety manual for instructions on how to handle liquid nitrogen safely. The Dewar supplied with the LNP95 has a safety release valve built into the siphon assembly. Always use in a well ventilated room.

Feedback

Your feedback will be greatly appreciated, please go to www.linkam.co.uk to fill in the Feedback form.

Safety Precautions

1) Read this guide before using the equipment. Save these instructions for later use.
2) Follow all warnings and instructions which may be placed on the programmer or stage.
3) If for any reason the mains fuse needs to be replaced then it must be replaced by one of the same type and rating as shown in the equipment ratings.
4) To prevent electric shock, do not remove the cover of the controller or associated electronics.
5) Never use the equipment if a power cable has been damaged. Do not allow any heavy objects to rest on the power cables. Never lay the power cables on the floor.
6) Do not obstruct any ventilation holes. Do not attempt to insert anything into these openings. Provide adequate ventilation of at least 75mm all around the equipment.
7) Do not expose the equipment to water. If for any reason it gets wet then unplug it from the mains and contact Linkam Scientific Technical Support.
8) The equipment is not intended to be used outdoors**.**
9) Each product is equipped with a 3-wire grounded (earth) mains plug or a free-end 3 wire mains lead. The plug only fits into a grounded-type outlet. The free-end mains lead should be connected to a correctly grounded 3-wire mains outlet. Do not defeat the purpose of the grounded (earth) type plug.

Free - end mains leads are colour coded as follows :

Colour	Function
Brown	Live
Blue	Neutral
Green/Yellow	Earth (Ground)

10) If any problems occur then unplug the equipment from the mains outlet and contact Linkam Scientific Technical Support.
11) Do not remove the cover from the equipment unless the mains inlet has been removed. Any servicing should be carried out by qualified service personnel.

Symbol References

Caution:

This safety symbol is on the back panel of the equipment and warns:-

The user must not make or remove any connections while the unit is powered on.
To avoid electric shock do not remove the cover. Refer servicing to qualified service personnel.

Caution:

This warning symbol indicates that the surface labelled with this symbol may be hot.

Introduction

Thank you for purchasing the FDCS196 Freeze Drying Stage system. Please take the time to read through the manual as it will help you to make the most out of the equipment.

FDCS196 System

FDCS196 Stage Specifications

Objective Lens WD:	4.6mm
Condenser lens WD:	12.5mm
Objective light approach angle:	115°
Maximum temperature:	125°C
Minimum temperature:	-196 °C
Maximum heating rate:	150°C/min
Maximum cooling rate:	60°C/min
Maximum cooling Rate under vacuum:	130°C/min
Maximum vacuum:	10^{-3}mBar
XY-Manipulators travel:	16mm
Vacuum flange type:	FK10 / FK10
Aperture hole:	1.3mm
Weight:	0.6Kg

FDCS196 System

The system consists of a FDCS196 stage, a T95-LinkPad System Controller, LNP95 liquid nitrogen cooling pump system, Pirani vacuum gauge and Linksys32 software.

The FDCS196 Pro System has an additional 1.5L Edwards Vacuum Pump and MV196 motorised vacuum valve control System.

Linksys32 System control software and digital video capture can be added as an option to control from PC.

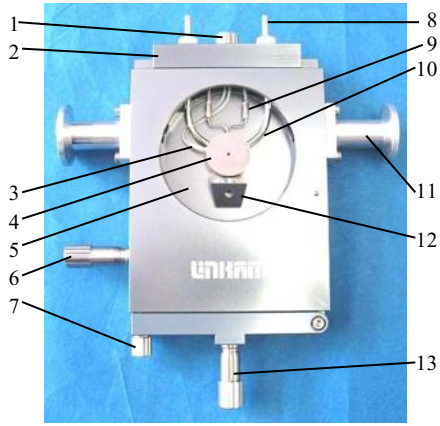
The FDCS196 stage is mounted onto the microscope by using specific stage clamps or an adaptor plate.

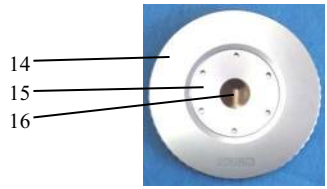
This manual will describe instruction for setting up a standard FDCS196 System.

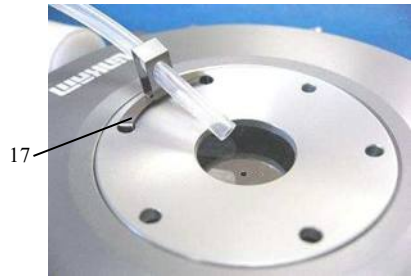
For the Pro System see page 13 and 21 to set up and control the MV196 Motorised Vacuum Valve System using Linksys32 software or the LinkPad.

Stage Anatomy

1. Lemo connector for Stage Connection Lead
2. Heating element carrier assembly
3. Platinum temperature sensor
4. 22mm Diameter copper heating block (nickel plated)
5. Sample chamber
6. X-Sample manipulator
7. Door locking thumbscrew
8. Liquid nitrogen cooling connector
9. Heating element wire
10. Stainless steel cooling tube
11. Vacuum port
12. Sample holder ramp
13. Y-Sample manipulator
14. Stage lid
15. Lid insert
16. Glass window (0.3mm thick)
17. Tube Clip Holder and tubing for Nitrogen de-fogging stage window







Mounting Stage to Microscope with Dovetail Substage

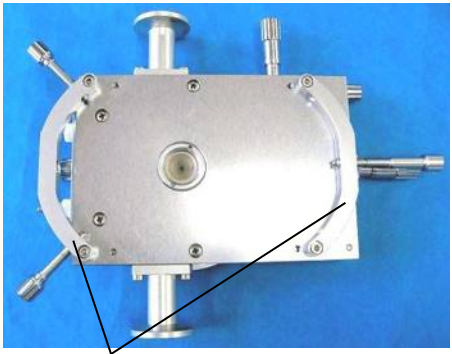
The following description is for mounting the stage on to microscopes which have a circular dovetail substage assembly.

Attach the curved stage clamps (part no. 9542) to the base of the stage using the supplied hex screws and the outer most holes in the base plate.

Adjust the two positioning screws so that approximately 5mm of thread is exposed on the inside edge of the clamp. This will roughly position the stage in the centre of the dovetail.

Place the stage onto the dovetail then focus a 10X objective lens on the aperture of the silver block. Using the two positioning screws ensure that the aperture is in the centre of the field of view.

For other types microscope's substage, see the included diagram for fitting instruction.



9542 curve clamps set

Dovetail substage

Setting up the Condenser for Koehler Illumination.

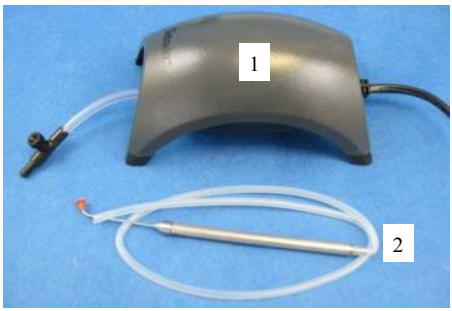
Place a small sample on a cover slip and place onto the surface of the silver block. Use the 10X lens to focus on the sample. Now close down the microscope field diaphragm and adjust the condenser focus so that the edges of the diaphragm are in focus. Now use the condenser positioning screws to centre the condenser in your field of view. Open the diaphragm so that it just fills the field of view.

For more information about Koehler illumination see the extremely informative 'microscopy Primer' on the Molecular Expressions website.

http://micro.magnet.fsu.edu/primer/index.html

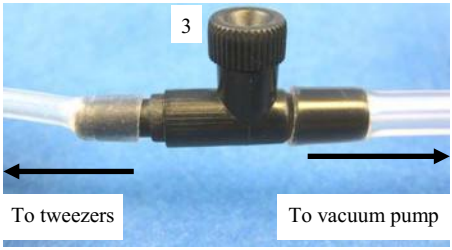
Vacuum Tweezers

The vacuum tweezers are used to manipulate the glass sample slides onto the silver block to prevent fingerprints on the glass and scratching the surface of the silver block when using standard fine tip metal tweezers.
The System is supplied with a Vacuum Tweezers Kit which consists of a Vacuum Pump (1) and tweezers (2).

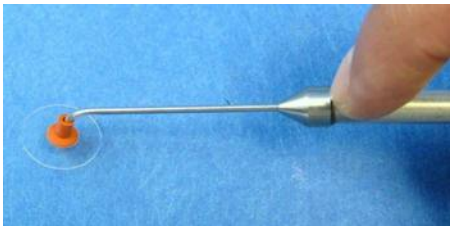


Connect the tubing at the end of the tweezers to the Regulator Valve (3) connection.

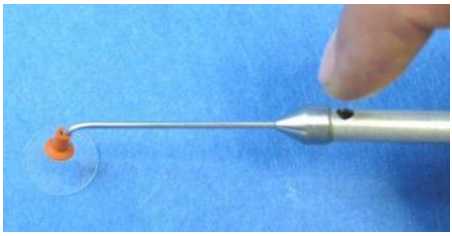
Use the dial on the valve to adjust the vacuum for the tweezers.



Use a finger to block the hole of the tweezers to pick up a sample cover slip with the suction cup.



Release the finger to drop the sample cover slip.



Connecting The Instruments

T95 System Controller
Cable Connections

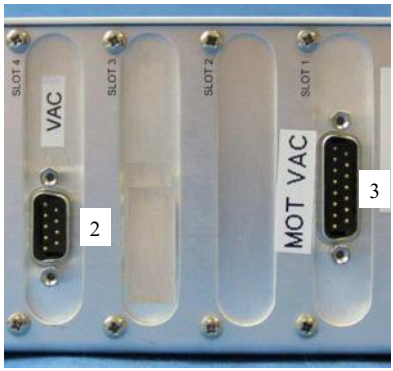
For more details on the T95 System Controller please refer to the T95 System Controller manual.

T95 back panel

Connect the Stage Cable to the Lemo Connector on the FDCS196 stage and connect the other end to the Stage Connection Socket (1).

Connect the cable from the Pirani Gauge to the Slot 4; labelled 'VAC' (2).

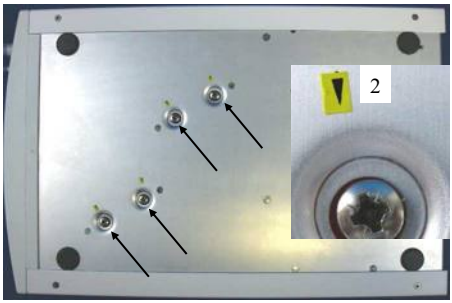
If you have got the Pro System with MV196 Motorised Vacuum Valve System, connect the cable from the MV196 Motorised Valve to Slot 1; labelled 'MOT VAC' (3).



LNP95 Cooling Pump Connection

Remove Transit Screws

Before using the LNP95 remove the 4 transit screws, marked by small yellow labels (2), from the base of the LNP95. Transit screws shown by arrows in the adjacent image. These screws hold the pumps in place for shipping.



Keep the screws safe by screwing them into the holes on the back panel as shown by the arrows.

The screws must be replaced back into the transit holes on the base, when shipping back to Linkam for service or repair.

Back Panel Cable Connection

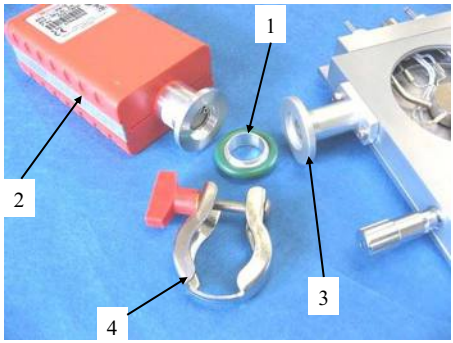
Connect the Instrument Bus Cable (3) between the LNP95 and T95 as shown.

Note: either of the purple coloured Instrument Bus Sockets on the LNP95 can be used.

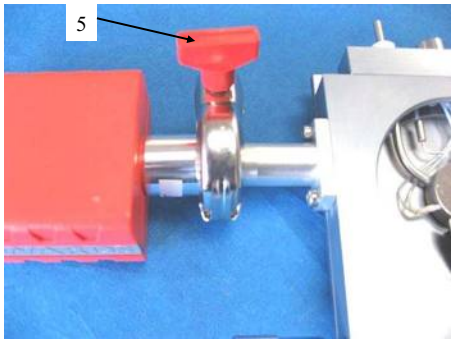
LNP95 MUST BE SWITCHED ON BEFORE T95 SYSTEM CONTROLLER

Connecting the Pirani Gauge

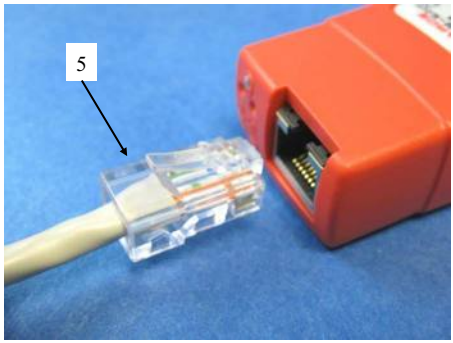
The surfaces where the O-ring (1) sits should be completely free of dirt. The O-rings should be smeared with a small amount of vacuum grease before loading in between the Pirani Gauge (2) and the Stage Vacuum Connector (3).



Ensure that the O-ring is correctly seated in the gauge and vacuum connector before tightening the Clamping Bracket (4) screw (5).

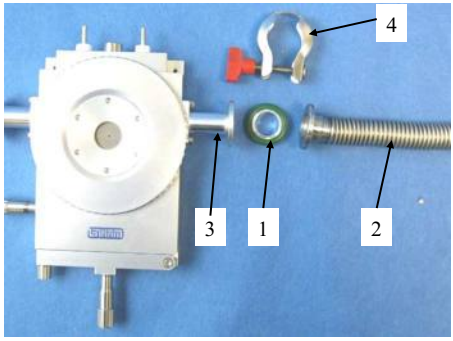


Connect the Pirani Cable with the LAN type connector (5) to the Pirani and the other end of the cable to the back of the T95; Slot 4; labelled 'VAC', see page 9.



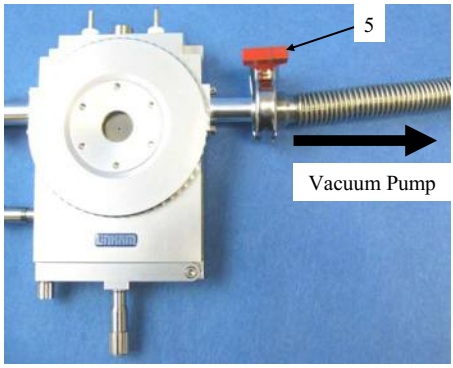
Flexible Stainless Steel Vacuum Tube Connection

The surfaces where the O-ring (1) sits should be completely free of dirt. The O-rings should be smeared with a small amount of vacuum grease before loading in between the Flexible Stainless Steel Vacuum Tube (2) and the stage Vacuum Connector (3).



Ensure that the O-ring is correctly seated in the Steel Vacuum Tube and vacuum connector before tightening the Clamping Bracket (4) screw (5).

Connect the other end of the Flexible Stainless Steel Vacuum Tube to a Vacuum Pump.



Note: the FDCS196 is only tested with a vacuum of 10^{-3}mBar. Do not use a vacuum lower than this.

FDCS196 Pro System Setup

Only read the following section if you have purchased the FDCS196 Pro System.

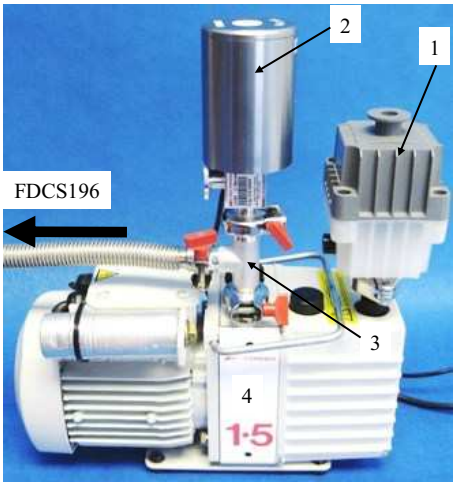
Edwards Vacuum Pump Setup

Please read the Edwards Vacuum manual to fill the pump with oil and to install the mist filter (1).

MV196 Motorised Valve System Set-up

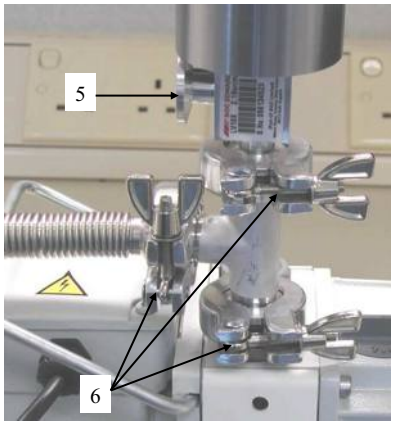
The MV196 Motorized Vacuum Control System consists of a motorized valve (2) connected to a T -piece (3) on top of the Edward Vacuum Pump (4).

Please note that the motorized valve accurately controls the vacuum within the stage but the maximum vacuum possible is not as great as when using a simple on/off style valve.



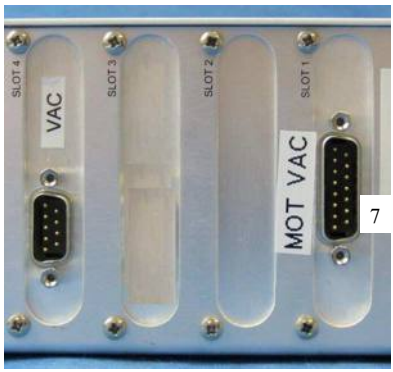
Mounting the MV196

The MV196 has an vacuum flange just like the vacuum ports on the sides of the FDCS196 Stage. Use a small amount of vacuum grease to coat a thin layer on the surface where the O-ring will sit. Coat the surfaces of the T-piece with vacuum grease as well. The valve has two ports, there is one in line and one protruding perpendicularly out of the side of this valve. The side port (5) is for the controlled vacuum vent and therefore should not have anything attached to it.



Use the Clamping Bracket (6) to secure the T-piece to the port as seen in the opposite picture. Secure the vacuum pipe between the stage and T-piece using the supplied O-rings and clamping rings (6).

Connect the cable from the MV196 Motorised Valve to Slot 1; labelled ‘MOT VAC’ (7).

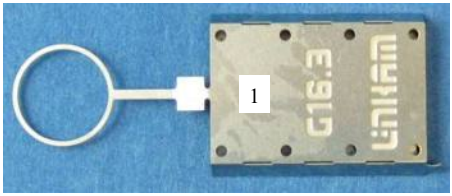


Sample Preparation and Loading

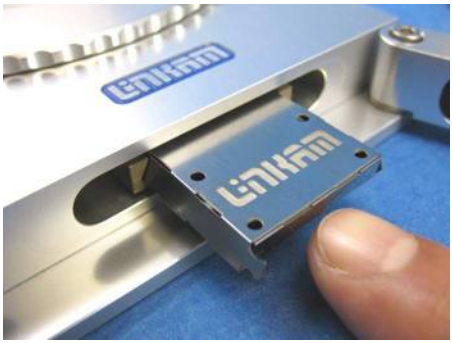
The stage chamber must first be prepared for vacuum. Ensure that the chamber is completely clean and dry inside. Finger grease and dirt must be cleaned off with IPA (isopropanol).

Sample Loading Using G16.3 Sample Holder

A Freeze Drying Sample is prepared using a G16.3 Sample Holder (1).



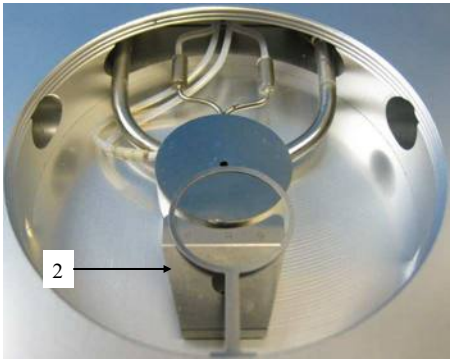
To load the G16.3 into the FDCS stage, open the door of the stage by unscrewing the door thumb-screw. The G16.3 slides into the brass housing located just inside the door.



The Sample Holder Ramp (2) will guide the G16.3 up on to the silver block.

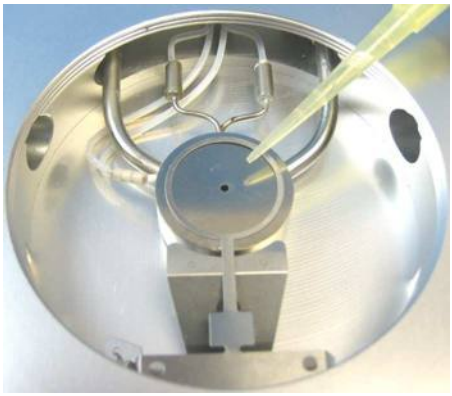
Close the door and tighten the thumbscrew to seal the stage.

Adjust the XY manipulators so the holder is in the middle of the silver block



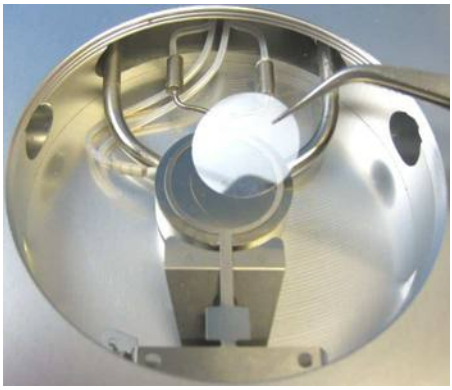
At standard atmospheric pressure the contact between the sample and the silver block is enough to establish good thermal contact as the air molecules in any minute scratches will conduct the heat, but when pulling a vacuum these are drawn out of the stage. Objective immersion oil (supplied) has the same refractive index as quartz and ensures an almost perfect thermal seal between block and crucible.

Using either the dropper or micropipette place a tiny drop of oil on the silver block. Be careful not to place the oil over the aperture hole of the silver block.

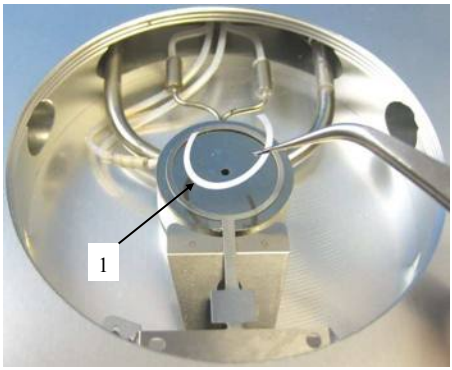


Use the supplied Vacuum Tweezers or a pair of tweezers place a clean W16Q, (16 x 0.3mm) Quartz Sample Window directly on top of the oil inside the G16.3 Sampler Holder.

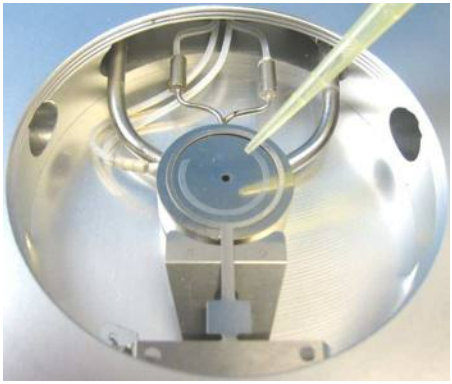
Adjust the XY manipulators by a few turns in both directions to spread out the oil underneath the Quartz Sample Window evenly.



Using a pair of tweezers place a Shim Spacer (1) in the middle of the Sample Holder.



Pipette 2µl of sample on the Quartz Sample Window inside the Shim Spacer.



Use the supplied Vacuum Tweezers or a pair of tweezers place a clean W13G, (13 x 0.17mm) Glass Sample Window on top of the sample, ensuring it touches the Shim Spacer.

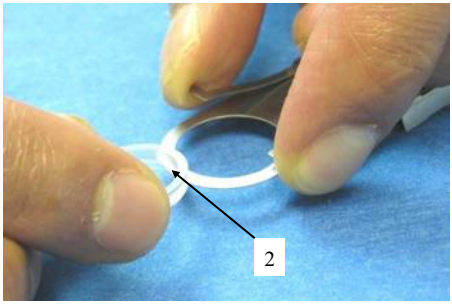
Use the XY manipulators to move the Sample Holder so the edge of the sample sits across the aperture hole of the silver block. This makes visual location of the sample easier once analysis starts.

Sample Loading Using FDCS/CC Crucible Carrier and Quartz Crucible

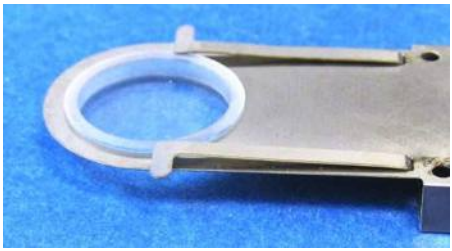
This method is used if you are preparing the sample away from the FDCS196 stage.

The crucible carrier has two spring clips to hold the crucible flat against the silver block. This is to ensure good thermal contact.

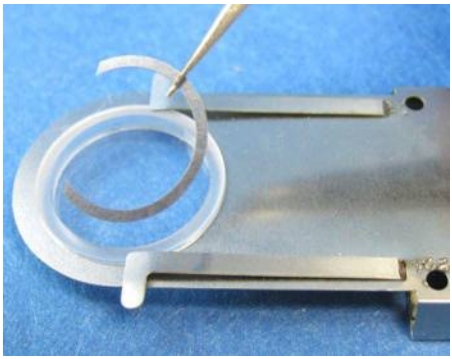
To load the quartz crucible into the carrier, lift the two spring clips about 2mm upward and slide the crucible in to the holder.



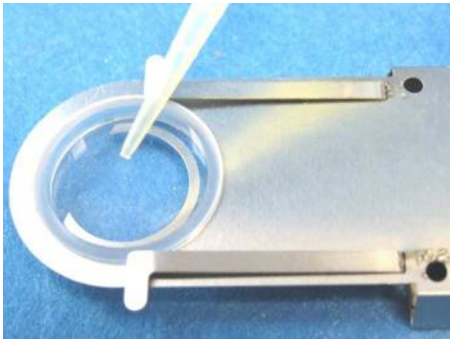
Release the two spring clips to hold the Crucible in place.



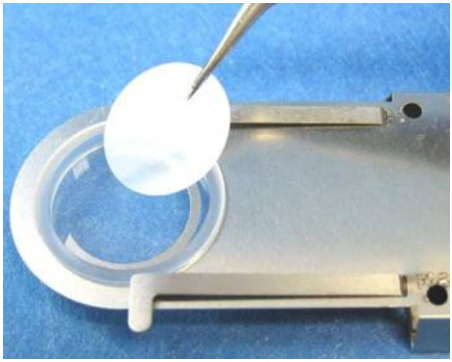
Use a pair of tweezers to place a Shim Spacer inside the Quartz Crucible.



Pipette 2µl of sample inside the Shim Spacer.

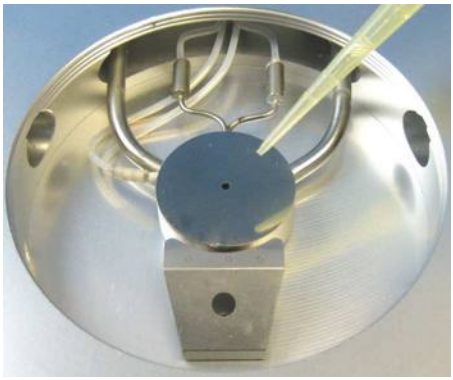


Use the supplied Vacuum Tweezers or a pair of tweezers to place a clean W13G, (13 x 0.17mm) Glass Sample Window on top of the sample, ensure it touches the Shim Spacer.



Before loading the Crucible Carrier the silver block must be prepared with silicon oil to provide a good thermal contact between the crucible and the silver block when the stage is under a vacuum.

Using either the dropper or micropipette place a tiny drop of oil on the silver block. Be careful not to place the oil over the aperture hole of the silver block.



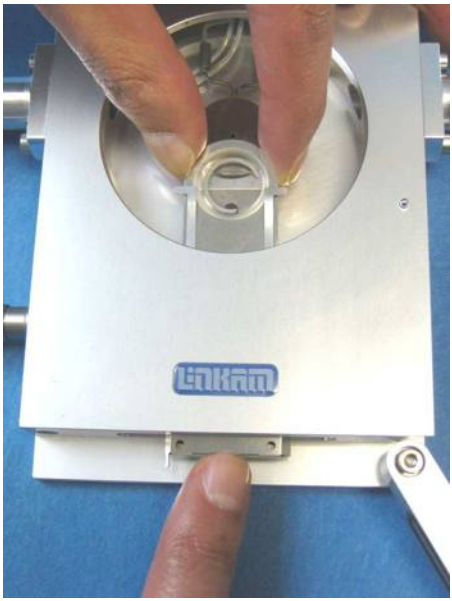
Load the Crucible Holder with the prepared sample into the FDCS196 stage. Unscrew the Y manipulator (the one screwed into the stage door) so that when you close the door, the FDCS/CC doesn't push the crucible right off the edge of the block and the silicon oil with it.

Open the door of the stage by unscrewing the door thumbscrew. The FDCS/CC slides into the brass housing located just inside the door. There is a Sample Holder Ramp just before the silver block which guides the crucible up on to the block.

With one hand push the FDCS/CC and with the other hand lift the edge of the crucible up the ramp and over the drop of oil. When the drop of oil is roughly in the centre of the crucible gently lower the crucible on the block. Now close the door of the stage and screw in the door locking screw. This screw only needs to be finger tight.

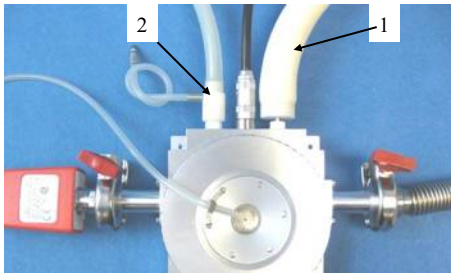
Adjust the XY manipulators by a few turns in both directions to spread out the oil underneath the Quartz Crucible.

Use the XY manipulators to move the FDCS/CC so the edge of the sample sits across the aperture hole of the silver block. This makes visual location of the sample easier once analysis starts.



Cooling Connections

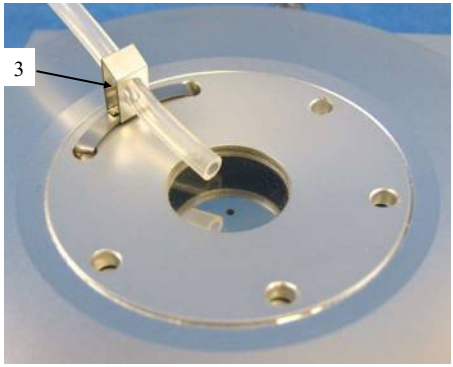
The Dewar Siphon (1) is the thick white foam tubing and is attached to the liquid nitrogen Dewar. The thin black capillary tube inside the white foam tube must be inserted into the liquid nitrogen cooling connectors on the stage.



The white tubing slides on to the outside of the connector. Twist the siphon whilst sliding it on and push until it comes to a stop. It does not need to go all the way to the base of the connector.

The thicker silicon tube from the LNP95 cooling pump ends in a white PTFE connector (2), this is pushed over the end of the other stainless steel connector as seen in the image.

The smaller tube from the outlet on the LNP95 should be placed in position on the top of the lid using the Tube Clip Holder (3). This tube blows warm recycled nitrogen gas across the lid window to prevent condensation on the viewing window surface.



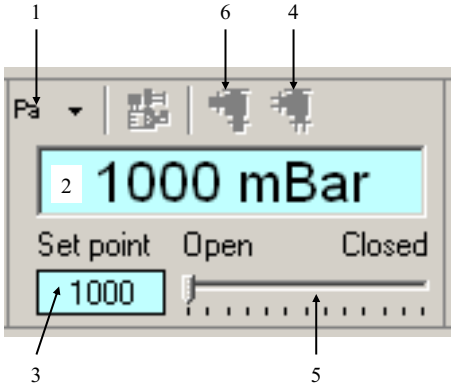
Using the FDCS196 System

Programming Temperature

Please refer to Linksys32 Software manual to program the temperature by software using a PC.

Using Linksys32 Software to Control MV196 Motorised Valve

When the Pirani gauge and / or MV196 System are connected correctly to the T95, you will see an additional Vacuum Toolbar in the Linksys32 software main window. See opposite picture.



To set vacuum units click on the Pa icon (1) to select Pascal's, or use the pull down list to select other units.

Set Vacuum

The Pirani gauge will display a live vacuum reading shown in the Main Vacuum Window (2).

To set a vacuum, turn on the Vacuum Pump and type in a vacuum value (value dependent on unit chosen) in the Set point's box (3). Click the Close Valve icon (4) and the motorised valve will automatically adjust itself to achieve the set vacuum value.

Note: a graphical indicator of the valve position between the Close and Open position can be seen in Motorised Vacuum Valve Indicator Bar (5).

Note: to take the FDCS196 back to atmospheric pressure, turn off the vacuum pump and click on Open Vale icon (6).

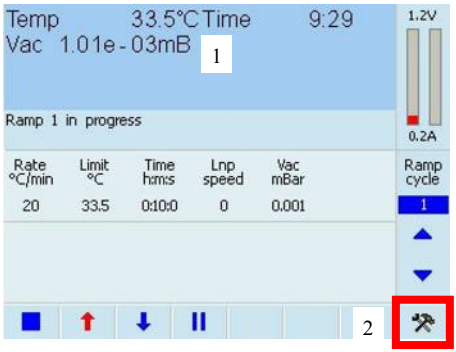
Note: if you have just the standard FDCS196 system with no MV196 system. The Open Valve and Close Valve icons are greyed out and will not function.

Using T95-LinkPad to Control MV196 Motorised Vacuum Valve

The T95-LinkPad works as a standalone unit. Please make sure Linksys32 software is not running.

With the Pirani Gauge connected you will see a live vacuum reading (1) on the Link Pad display.

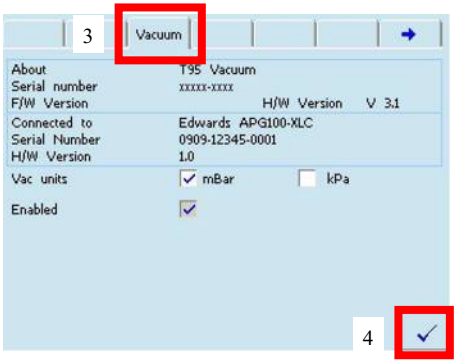
On the display mB represents mBar (millibar) unit or kPa (kilopascals) unit.



To change the vacuum unit, touch the Setup icon (2) to go to the Setup Tab page.

Touch the Vacuum Tab (3) to display the Vacuum Setup menu. Touch either the mBar or kPa check box to select the vacuum unit.

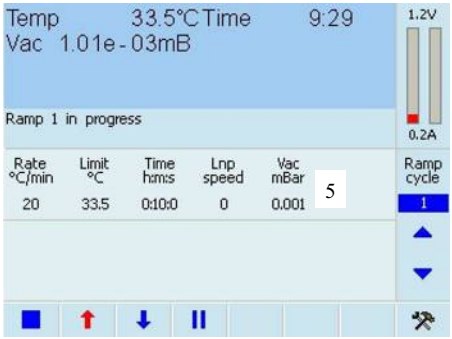
Touch the Confirm icon (4) to go back to the main screen.



If you have the FDCS196 Pro System with the MV196 Motorised Valve connected there is an extra column labelled 'Vac mBar / kPa' displayed in the main screen (5).

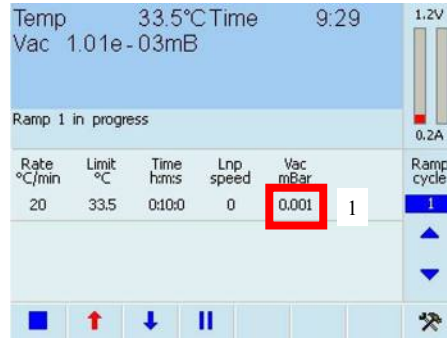
This option is available for you to program in a vacuum value for each Ramp in the Programmable Parameters.

Note: please refer to the T95-LinkPad manual for programming the temperature using LinkPad.



Vacuum Parameters

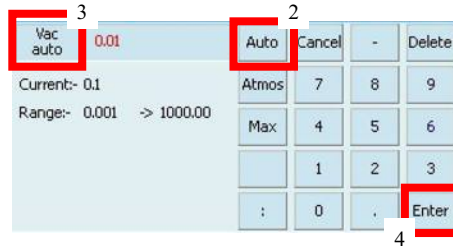
Touch the active area (1) to change the main screen to the Vacuum menu screen.



In the Vacuum main screen there will be 3 options to control the Vacuum value.

1. Auto: in this mode the motorised valve will automatically adjust itself to your programmed vacuum value.

Touch the **Auto** button (2) and make sure the word 'auto' is displayed under the word 'Vac' (3). Use the keypad to type in a vacuum value and touch **Enter** button (4) to go back to the main screen.



Note: The MV196 is only designed to hold a controlled vacuum between 100mbar to 0.1mbar. Any values outside this range may not be accurate.

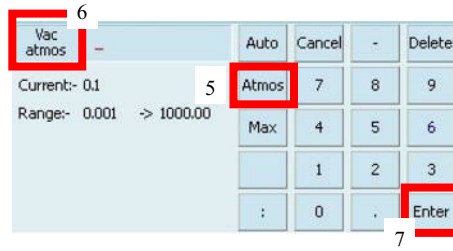
Note: touch the **Cancel** button to go back to the main screen without changing the vacuum value.

Note: Current:- is the current programmed rate.

Note: Range:- is the maximum and minimum vacuum value that can be program for the selected vacuum unit (mBar or kPa).

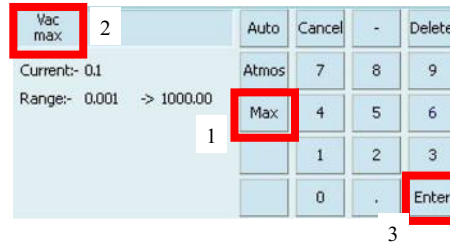
2. Atmos: in this mode the motorised valve will fully open to set the vacuum pump to atmospheric pressure.

Touch the **Atmos** button (5) and make sure the word 'atmos' (6) is displayed under the word 'Vac' to set the Ramp to **Atmos** mode. Touch **Enter** button (7) to go back to the main screen.



3. Max: in this mode the motorised valve will close fully to get the maximum vacuum from the vacuum pump.

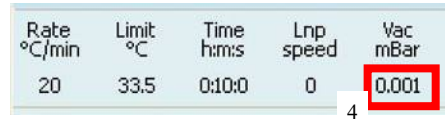
Touch the **Max** button (1) and make sure the word 'max' (2) is displayed under the word 'Vac' to set the Ramp to **Max** mode. Touch **Enter** button (3) to go back to the main screen.



Running a Profile with Vacuum

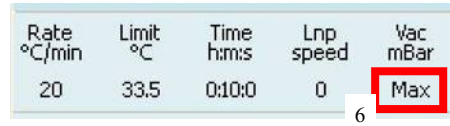
When a Profile is running the following display in the main screen will help you to visualise the programmed vacuum setting in the **Ramp**.

1. The programmed vacuum value will be shown (4) if the Vaccum is set to **Auto**.



2. The word 'Atmos' (5) will be shown if the Vauum is set to **Atmos** mode.

3. The word 'Max' (6) will be shown if the Vacuum is set to **Max** mode.



Note: when a Profile is running and there is no Vacuum setting in the Ramp, the current Ramp will use the vacuum setting of the previous Ramp.

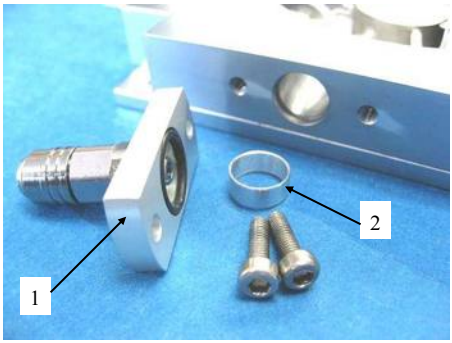
Appendix

Using The Stage With Gas Connectors

Installing Gas Connectors

This is used when vacuum is not needed.

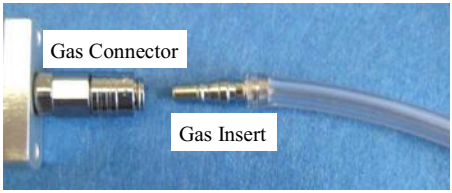
To replace the vacuum port with a gas port connector (1), use the hex key provided to remove the two screws that hold the vacuum port in place.
Do the reverse to fit in the gas port connector and make sure that the vacuum/gas seal (2) insert is placed between the stage and connector.



The Gas Connector is self closing, it is open when the Gas Insert is pushed into the Gas Connector.

To close the connector, the Gas Insert is removed.

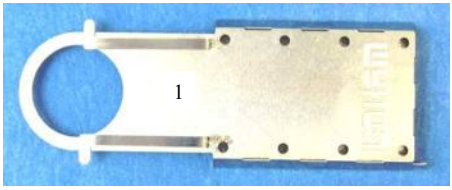
To use the Gas Insert, connect one end to a tube (3mm inner diameter, 6mm outer diameter) and push the other end into the Gas Connector. You should hear a “click” when the two parts are connected properly.



To remove the Gas Insert, just push back the outer sleeve of the Gas connector toward the base of the FDCS196 Stage and the Gas Insert should drop out.

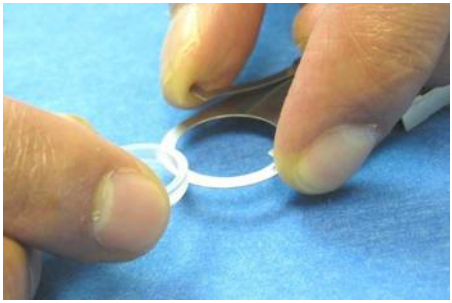
FDCS/CC Crucible Carrier and Quartz Crucible

By using the FDCS/CC sample carrier (1), sample can be loaded into the stage without removing the stage lid. The crucible carrier is loaded through the side door.



The crucible carrier has two spring clips to hold the crucible flat against the silver block. This is to ensure good thermal contact.

To load the quartz crucible into the carrier, lift the two spring clips about 2mm upward and slide the crucible in to the holder.



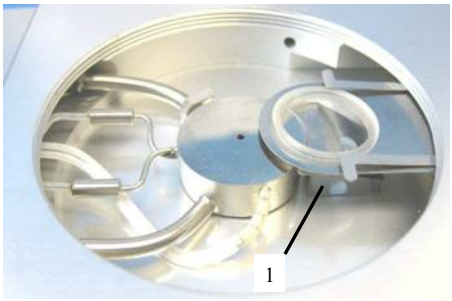
Release the two spring clips to hold the Crucible in place

Load the sample into the quartz crucible. Use as little sample as possible to ensure a small thermal load and therefore better temperature sensitivity.

Microscope objectives require a flat surface to give maximum field of view. Place a 13mm coverslip on top of your sample and gently tap the top with the back of the vacuum tweezers to disperse powder samples or ensure good thermal contact of irregularly shaped samples.

Open the side door of the stage by unscrewing the thumbscrew and carefully push the FDCS/CC and crucible into the XY slide mechanism.

The crucible will be guided to the surface of the silver block by the Sample Holder Ramp. Close the door and tighten the thumbscrew to seal the stage.



Cooling Connections

These connections need only be made if the experiments are to be carried out below room temperature.

The Dewar siphon (1) is the thick white foam tubing and is attached to the liquid nitrogen Dewar. The thin black capillary tube inside the white foam tube must be inserted into the liquid nitrogen cooling connectors on the stage.
The white tubing slides on to the outside of the connector. Twist the siphon whilst sliding it on and push until it comes to a stop. It does not need to go all the way to the base of the connector.

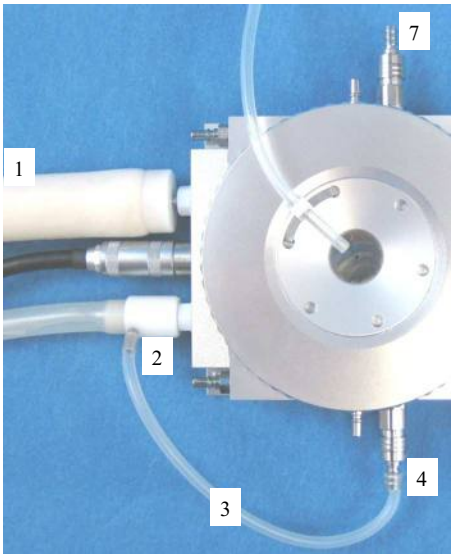
The thicker silicon tube from the LNP95 cooling pump ends in a white PTFE connector (2), this is pushed over the end of the other stainless steel connector as seen in the image.

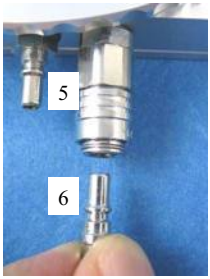
The short tube branching from the side of this white connector is the Gas Purging Tube (3). There is a valve opening Insert connector (4) inserted into the end of this tube. During the purging procedure, insert this connector into the Gas Purge Valve (5) on the side of the stage to open it.

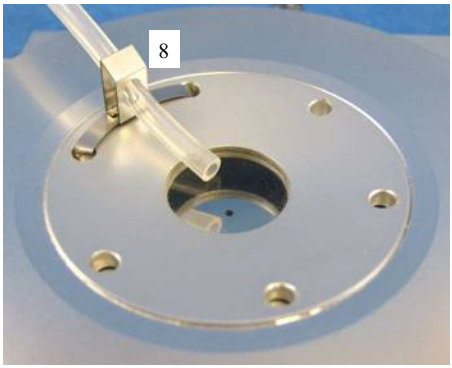
The Gas Purge Valve (5) is opened when the Gas Insert (6) is pushed firmly into the connector, a "click" is heard when the two parts are connected properly. To remove the Gas Insert, push the outer sleeve of Gas Purge Valve toward the stage and the Gas Insert (6) should drop out.

There is a second Gas Purge Valve on the opposite side of the stage to allow the gas to leave the stage. A Gas Insert must also be inserted into this Gas Purge Valve (7) when purging.

The smaller tube from the outlet on the LNP95 should be placed in position on the top of the lid using the Tube Clip Holder (8). This tube blows warm recycled nitrogen gas across the lid window to prevent condensation on the viewing window surface.







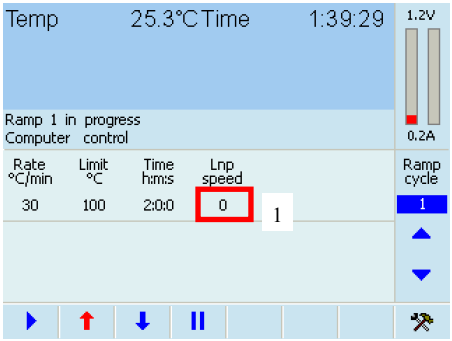
Purging Procedure

Before starting a cooling experiment, you will need to purge air from the stage chamber with dry nitrogen. This will remove the water in the air which would otherwise condense and freeze on the sample disrupting your image quality.
Before you can start purging, the LNP95 must be set to manual mode.
You can either use the LinkPad touch screen or Linksys32 software.

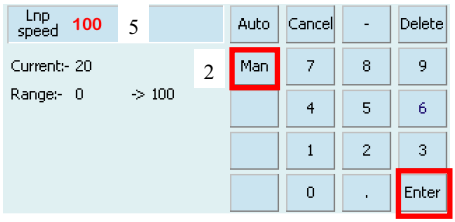
Use LinkPad to set the LNP95 to Manual Mode

Touch the active area (1) under 'Lnp Speed' to change to the LNP Screen Menu. Touch 'Man' (2) to switch the LNP95 to manual mode.

The word 'auto' is changed to 'speed' to indicate the LNP95 is functioning in manual mode.



Using the Keypad type in 100 (max pump speed) and touch the 'Enter' button (4) to start the LNP95 at the programmed speed (5).



Filling the Liquid Nitrogen Dewar

Please follow your health and safety manual for directions on how to handle liquid nitrogen and ensure that you have the correct safety equipment including gloves and safety goggles.

Fill the Dewar until approximately 2/3 full and replace the lid with siphon attached.

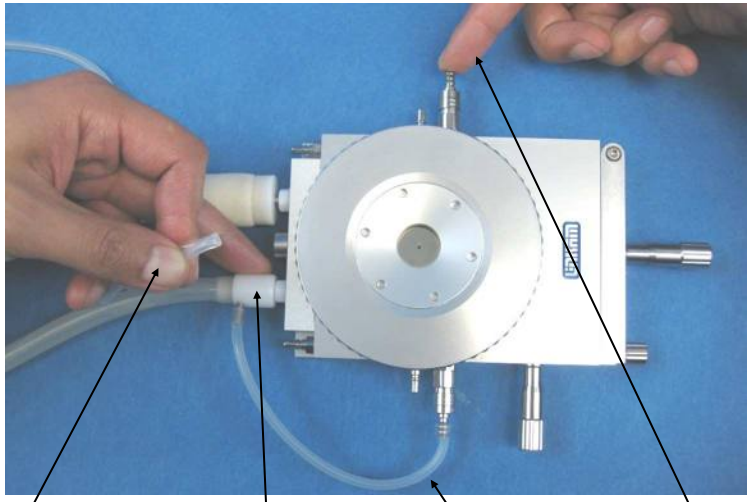
DO NOT FASTEN THE CATCHES.

Wait for the nitrogen to stop bubbling before fastening the catches.

Purging the Stage Method 1

There are two methods for purging the stage. Method 1 uses recycled nitrogen gas produced by the LNP95 from the Dewar.

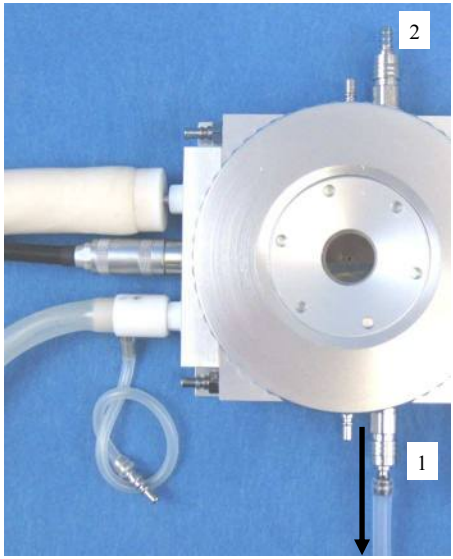
1. Make sure the stage lid is in place and the stage door is closed.
2. Switch on the temperature programmer and set the limit to 40°C. Press the START button and wait until the temperature limit is reached. Press HOLD to hold the temperature at 40°C.
3. Switch on the LNP95 cooling system and set it to manual mode, (see page 29) and set the speed to maximum of 100.
4. Check that the Gas Inserts are locked into place
5. Using a finger on the left hand, block the hole in the white plastic pump connector found on the perpendicular side to the purging tube. Still working with the left hand, pinch the narrow window tube to block it. This action will divert all of the nitrogen gas to the Purging Tube and through the Stage Chamber.
6. With the nitrogen gas flowing through the Sample Chamber, use a finger on the right hand to block the gas outlet for a few seconds to allow pressure build, then release the gas . Do this for a few minutes to purge the stage.
7. Look at the change of reflection in the stage window as the stage is pressurised and released to check that the stage is properly sealed. If there is no change, there may be a leak due to incorrect placing of the silicon o-rings in either the bottom or lid window or the window (top/bottom may be broken).
8. The purging procedure allows mixing of nitrogen gas with the residual air inside the Sample Chamber. By pressurising the chamber with nitrogen gas and releasing it, the air inside the Chamber is being diluted with the nitrogen gas.
9. Remove the two Gas Inserts and unblock the pump connector and window tube.
10. Change the LNP95 to **AUTOMATIC** mode so that the T95 automatically controls pump speed during your cooling experiment
11. Go to www.linkam.co.uk and register your equipment to see videos of how to purge and more.



Purging the Stage Method 2

This method uses an inert gas from a gas cylinder to purge the stage at temperatures above ambient when the LNP95 is not required.

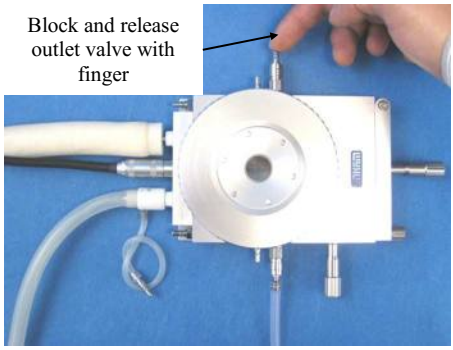
1. Make sure the Stage Lid is in place and the Stage Door is closed.
2. From a gas cylinder connect the Gas Insert with a tubing 3mm inner diameter and 6mm outer diameter to the Gas Purge Valve (1).
3. Connect a Gas Insert to the opposite side Gas Purge Valve (2).
4. Use the gas regulator to set a gas flow rate of 1.5L/min.
5. With the gas flowing through the Sample Chamber, block the gas outlet for a few seconds and releasing the gas outlet valve with a finger. Do this for a few minutes to purge the stage.



To gas cylinder

Reduce the gas flow rate to 20cc/min to continuously purge the stage or remove the two Gas Inserts to keep the chamber under closed inert atmosphere.

Note: Helium gas is not recommended for continuous purging. This gas has a very high thermal conductivity and will cool the silver heating block too much during an experiment and may cause the temperature to fluctuate.



Window Assembly

Lid Window Assembly

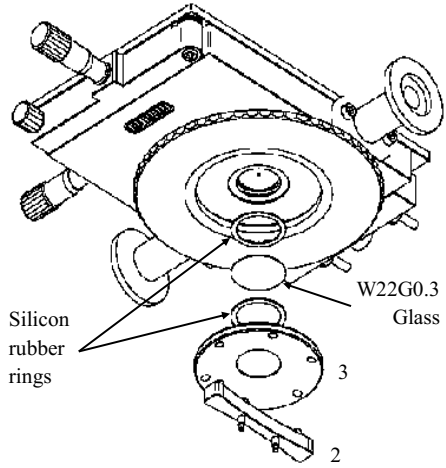
To replace the windows in the Stage Lid (1) use the Window Tool (2) and align the two wide spacing pins to the Tube Clip Holder holes and unscrew the Lid Insert (3).

The Stage Lid and Lid Insert should be turned upside down as shown in the diagram opposite and reassembled in the order indicated.

The Lid Insert should be screwed down until the cover slips are held firmly, then turn the assembly over and screw down the Lid Insert until it is felt to come to a stop.

Note: W22G0.3, (22mm diameter and 0.3mm thick) glass window must be used for when the stage is to be used under vacuum.

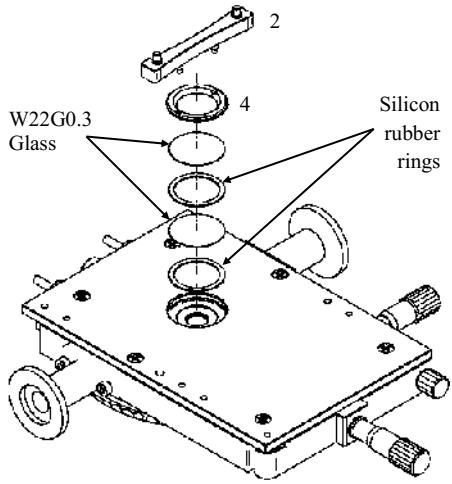
Note: you can use the W22G (22 x 0.17mm) window if the stage is used without a vacuum.



Bottom Window Assembly

Use two narrow spacing pins of Window Tool (2) to align it to the two holes of Window Locking Ring (4) and unscrew.

Reassemble the bottom window as shown in the opposite diagram



Spares and Accessories

These spares are organised into convenient kits. Purchase a spares kit to avoid downtime with your stage and eliminate future shipping costs.

The FDCS196 heating element is extremely durable if used carefully. However, it is made from pure silver which is a soft metal. It can be easily scratched, which will compromise the heat flow to the sample and reduce accuracy. The platinum temperature sensor is brittle and can be broken if cleaning is not carefully performed. We recommend a spare heating element to avoid downtime with your stage while element is being repaired.

Part No.	Part Name	Part Description
7503	FDCS Kit	Full Replacement Spares Kit
	WG	Water/Gas Valve Insert x2
	WVC	Water/Gas Valve Connector x2
	SSR	Silicon Rings for Lid and Base (Set of 4)
	RI17	Stainless Steel Ring Set
	THC	Tube Clip Holder (for Nitrogen de-fogging stage lid tube)
	ORTHMS	Set of O-Rings for THMS Stage Body and Lid
	THMS/Q	15mm diameter Quartz Crucible for FDCS/CC
	FDCS/CC	Crucible Carrier with Spring Clips for FDCS196
	G16.3	16.3mm Sample Carrier
	W22G0.3	22mm diameter Glass Lid/base Window (0.3mm thick) Box of 50
	W16G	16mm diameter Glass Sample Window (0.17mm thick) Box of 100
	WT	Window Tool (for unlocking lid insert and base locking ring)
	FDCS SP	Shim Spacer for Sample Prep in FDCS (OD:14mm x ID: 12mm x 70 micron) x40
	SO	Silicon Oil (1/4 fl.oz)
	W16Q	Quartz Sample Window (0.3mm thick) for W16G x4
	W13G	13mm diameter Glass Sample Window (0.17mm thick) Box of 100

Part No.	Part Name	Part Description
7504	FDCS Spare Windows Kit	Spare windows for Lid, Base and samples
	W16G	16mm diameter Glass Sample Window (0.17mm thick) Box of 100
	W22G0.3	22mm diameter Glass Lid/base Window (0.3mm thick) Box of 50
	SRR	Silicon Rings for Lid and Base (Set of 4)
	FDCS SP	Spacer for sample prep in FDCS (OD: 14mm x ID: 12mm x70 micron x40
	SO	Silicon Oil (1/4 fl.oz)
	W16Q	Quartz Sample Window (0.3mm thick) for W16G x4
	W13G	13mm diameter Glass Sample Window (0.17mm thick) Box of 100
	THMS/Q	15mm diameter Quartz Crucible for FDCS/CC

Part No.	Part Name	Part Description
9584	FDCSB	Spare Pure Silver Heating Element incl. Platinum Temperature Sensor

Part No.	Part Name	Part Description
18006	VP	E2M1.5 Edwards Vacuum Pump (including - EMF3 Mist Filter)

Part No.	Part Name	Part Description
7505	VacC-K	Spare Vacuum Connection Kit
	NW10-EB	NW10 Elbow 90 degree
	NW10-O	10mm bore vacuum O-ring
	NW10/16-O	10 to 16mm bore vacuum O-ring
	NW16-FH	500mm stainless steel flexible hose
	NW16-O	Clamping ring x3
	NW16-CR	Clamping Ring
	VG	Vacuum grease

Troubleshooting

Cooling fault diagnosis

Ensure that all connections to the stage and Dewar are as described in the specific manual and that the stage lid and top windows are properly sealed.

1. *The cooling rate is less than programmed.*
   There can be several causes of this problem, the most likely being that one of the connectors has become blocked or damaged. Check that each tube is fitted tightly to the connector and that none of the tubing is twisted or has come lose. The larger diameter tube leading from the LNP95 consists of a tube within a tube, check that the internal tube is connected, it may have come loose. Any constrictions of either the tubing or the connector will have a drastic effect on the cooling ability of the LNP95. If the connectors and tubing are OK, check that the capillary tubing to the Dewar flask is not bent or damaged and that the filter is intact and unblocked. If any damage has occurred to any of these items then it will be necessary to replace them. If no damage is found, check that the silver block is not constricted. This can be checked, simply by blowing through one of the steel cooling tubes using a compressed air line.

2. *Stage will not cool down to -196°C.*
   Check that the stage lid is not touching the silver block when screwed down. Check that the silver block has not been pushed down so that it touches the base of the stage. Check the sample holder ramp is not touching the silver block. Any of these faults will cause a substantial loss of cooling ability.

3. *Condensation and ice forming on the upper side of window*
   Realign the window gas tube clip to the required position in the stage lid.

4. *Condensation on the sample and/or the underside of lid window*
   This is due to the stage not being sealed properly and therefore allowing moisture in during purging or cooling. Check that the lid and bottom window are sealed correctly and that the silicon seals are in position.

Please visit www.Linkam.co.uk for more FAQ for the stage and instruments.

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