

**Linkam Scientific  
Instruments Ltd**

**DSC 600  
Stage Manual**

User's Guide



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## Important Notice

Please check that your Linkam equipment has not been damaged during transport. 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.

Please contact Linkam for custom modifications for specific applications.

In order to use this equipment successfully, please take time to read this manual all the way through before starting to work.

## 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

The programmer does not require any regular maintenance. If for any reason it is necessary to check the electronic calibration then a set of standard resistances can be supplied, which simply plug into the programmer in place of the stage, and indicate known temperature values. The standards are traceable to NAMAS (National Accreditation of Measuring and Sampling)

Before cleaning the case or front panel of the programmer, remove the mains lead from the wall outlet.

Use a small quantity of isopropyl alcohol with a soft cloth and gently wipe the surface.

## Regulatory Compliance Statement

This CI94 has been tested and found to comply with the following European directives :

- 1) EMC Directive 89/336/EEC as per
  - EN 50081-1:1992**
    - EN 55 022 A.C.Mains 150 KHz to 30 MHz
    - EN 55 022 Radiated Emissions 30MHz to 1GHz
  - EN 50082-1:1992**
    - IEC 801-2:1991 / prEN55024-2:1992 - 3 kV CD,8 kV AD
    - IEC 801-3:1984 / prEN55024-3:1991 - 3 V/m
    - IEC 801-4:1988 / prEN55024-4:1992 - 1 kV Power Lines
- 2) Low Voltage Directive (Safety) 73/23/EEC as per EN 61010-1 1993 and EN 61010-2 : 1994

## CI 94 Technical Specification

Stages using a platinum resistor sensor

Temperature Range: -196°C to 600°C (dependent on Stage)  
Temperature Stability:  $\pm 0.1^\circ\text{C}$  over the operating temperature of  $10^\circ$  to  $40^\circ\text{C}$   
Set Point Resolution:  $0.1^\circ\text{C}$   
Temperature Display:  $0.1^\circ\text{C}$  or  $0.01^\circ\text{C}$  resolution (switchable)  
Control Stability: All stages stable to  $\pm 0.1^\circ\text{C}$   
Temperature Sensor: Pt100 1/10 DIN or 0.01 Ohm at  $0^\circ\text{C}$   
Temperature Accuracy:  $\pm 0.1^\circ\text{C}$

Display: 2 rows of 24 characters, 5.5mm high LCD display with backlight  
Dimensions: 230 x 80 x 310 mm  
Weight: 1.9 Kg  
Operating Environment:  $5 \sim 40^\circ\text{C}$   
80% relative humidity at  $31^\circ\text{C}$  decreasing linearly to 50% at  $40^\circ\text{C}$  (without condensation)

## CI 94 Equipment Ratings

A.C Mains Supply: 90 - 264 ~  
A.C Frequency: 47 - 63 Hz  
Max Current: 2A  
Fuse: 2.5A (F) 250V~ Fuse must be replaced by one of the same type and rating

Dimensions: 230 x 80 x 310 (deep) mm  
Weight: 1.9 Kg  
Operating Environment:  $5 \sim 40^\circ\text{C}$   
80% relative humidity at  $31^\circ\text{C}$  decreasing linearly to 50% at  $40^\circ\text{C}$  (without condensation)

## 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.  
The equipment is not intended to be used outdoors.
- 8) 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) |

- 9) If any problems occur then unplug the equipment from the mains outlet and contact Linkam Scientific Technical Support.
- 10) 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 Reference



**Caution -**

This safety symbol is on the backpanel 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.

## Optical Information

It cannot be stressed enough, how important it is to have the microscope set up correctly, especially when working at high magnifications.

The following few paragraphs detail the setting up of the most important features of the microscope such as the light source and condenser lens.

To help explain the set up procedures for the microscope, a simple line drawing showing the essential parts of the microscope's optical path is shown below.

If the condenser working distance is less than 12.5 mm, we can supply a condenser extension lens for most reasonably new microscopes.

### Phase Contrast

Please contact Linkam for further details if you are using the stage with phase contrast.

### Light source centring and focus

The adjustment of the light source is only necessary when the microscope is first installed or when the bulb is replaced, provided that the adjustments made are not disturbed.

1. Open the field diaphragm on the base of the microscope to its largest extent and place a flat, thin piece of paper over it.
2. Turn the light source to maximum output.
3. Slide the lamp housing backwards and forwards until the image of the bulb filament is visible on the paper.
4. To centre the filament, adjust both the lamp vertical centring ring and the lateral centring screw on the lamp housing.
5. Pull the lamp housing slightly forward until the filament image is diffused, or alternatively fit a diffusing filter.

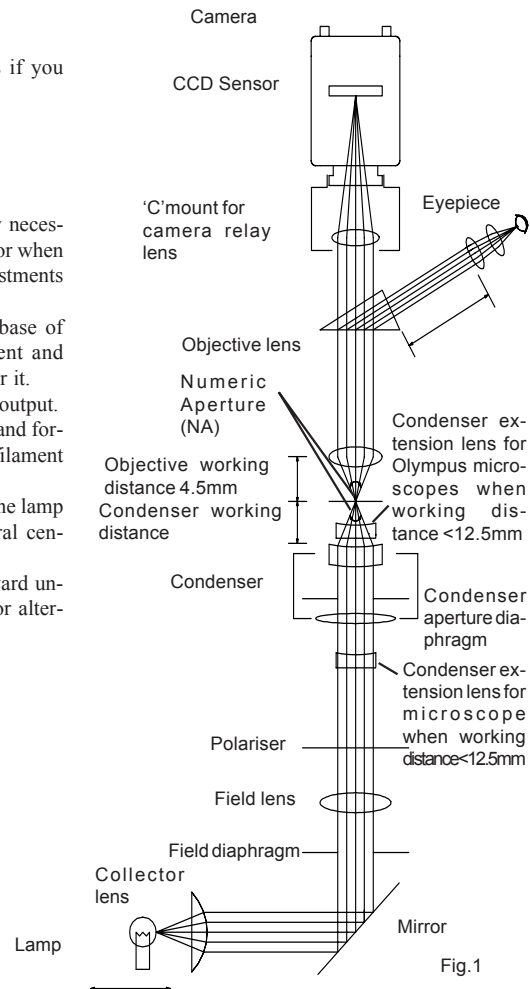


Fig.1

### Centring the condenser lens

It is extremely important to have a focused, centred and condensed light beam at all times and to ensure that the field diaphragm and condenser aperture diaphragm are set correctly.

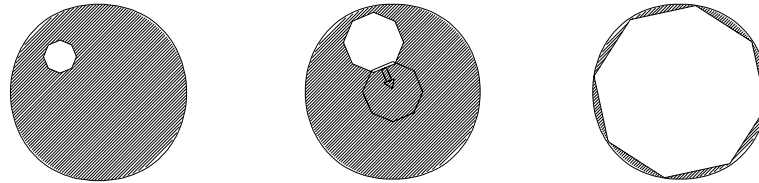


Fig.2

- 1) Select the 10X lens and close the field diaphragm on the microscope base to its smallest size. Rotate the condenser focus knob, moving the condenser up and down until a sharp image of the field diaphragm is seen clearly in the eyepiece.
2. Using the two condenser centring screws bring the field diaphragm image into the centre of the field of view.
3. Adjust the field diaphragm so that the image of the diaphragm is about the same as that of the field of view. If it is still not centred use the condenser centring screws again.

### Use of field diaphragm

The field diaphragm controls the diameter of the illuminated area on the specimen surface in relation to the field of view of the microscope. By stopping down the field diaphragm until it is slightly larger than the field of view, will reduce stray light, and in turn will increase image definition and contrast.

### Use of condenser aperture diaphragm

This adjusts the numerical aperture (N.A) of the illuminating system of the microscope and determines the resolution, contrast and depth of focus.

In general it should be stopped down to 70~80% of the numerical aperture of the objective lens for best overall results.

After completing focus adjustment, remove one of the eyepieces and look into the empty eyepiece tube. Adjust the size of the diaphragm, observing the image of the diaphragm which is visible on the bright circle of the objective exit pupil.

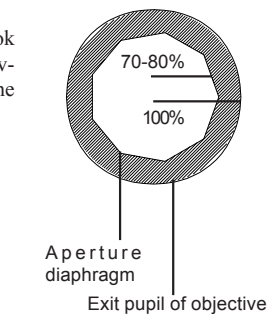


Fig.3

## **Mounting the DSC Stage on the Microscope**

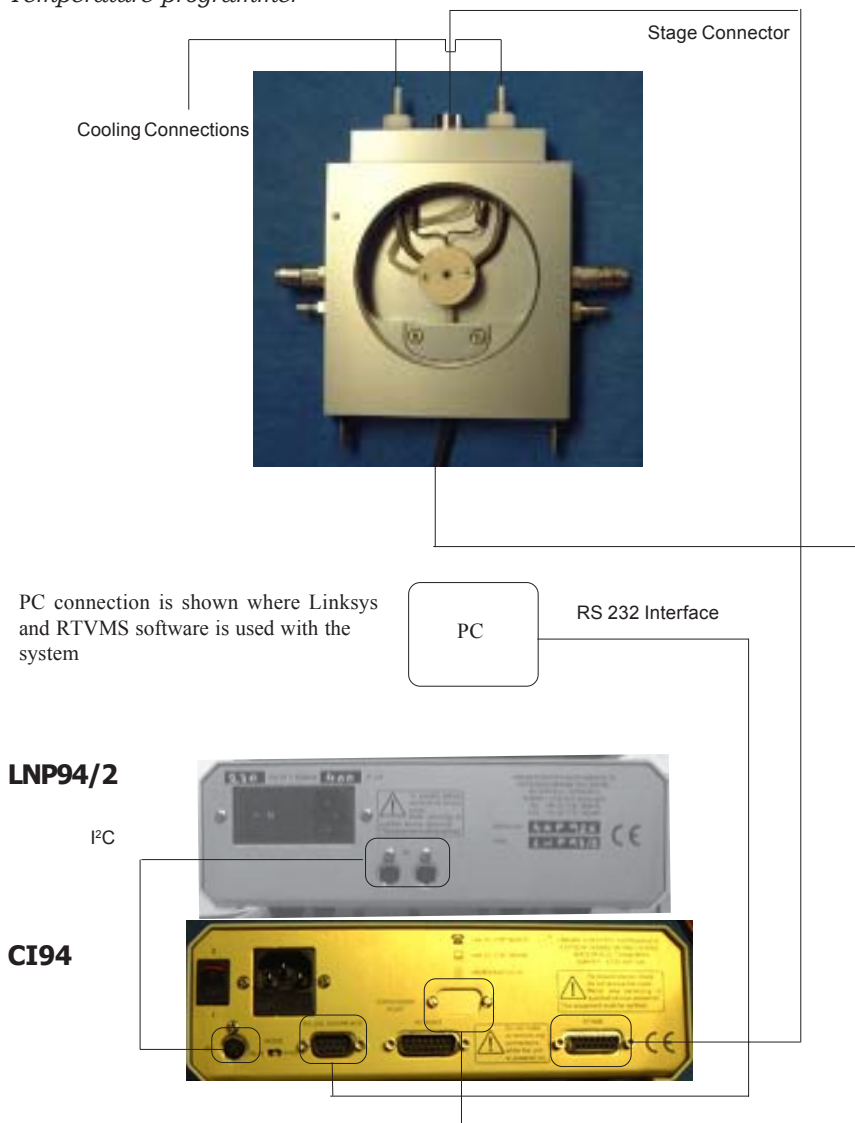
The DSC stage does not have its own set of clamps as seen with other Linkam stages. The best way to fix the stage to the XY table of the scope is with double sided adhesive tape. Microscopic clamps will become available in the near future.

Apply a couple of strips of tape to the underside of the stage and then mount to the microscope table. It is then possible to center the aperture in the silver block using the XY manipulation of the stage.



## Connecting the Instruments

Connecting the DSC600 to the PC, LNP94/2 and CI94 Temperature programmer



## Equipment Operation

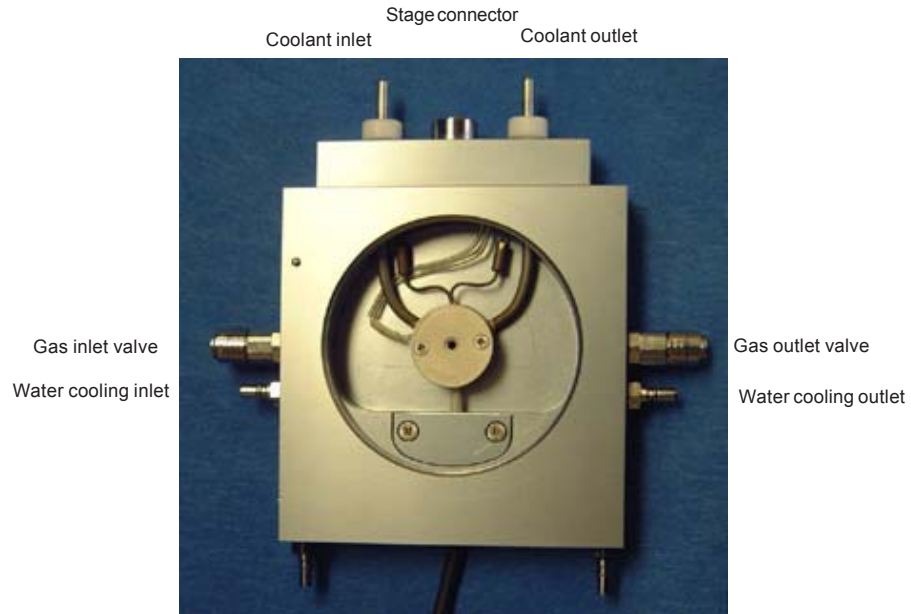
In order to avoid problems with the operation of the equipment, it is important to switch the units on in a recommended order.

When all the equipment is connected, the PC must be switched on first, then the programmer, pump (If cooling is required) and finally camera (if connected).

**Note: All equipment should be connected before anything is switched on.**

**The (LNP) liquid nitrogen pump is fitted with Transit screws which are there to hold internal pumps in place during transit to prevent internal damage. Remove these screws before using the pump. They are marked with yellow and black labels.**

## Connecting the LNP94/2 to the DSC600



### Setting up for cooling

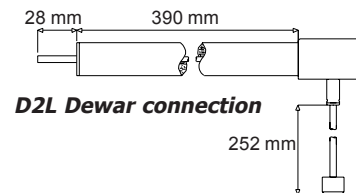
#### LNP94

The LNP cooling system can be used in conjunction with the DSC 600 stage to cool a sample down to below  $-194^{\circ}\text{C}$ . The software automatically selects the manual mode of cooling which requires you to input a pump speed into the toolbar of the software.

The pumpspeed window is labelled 'LNP'.

A small 2 litre desktop dewar flask with the fitted tubing (D2L) is supplied with the LNP.

**Note. Under no circumstances should the tube length be changed!**



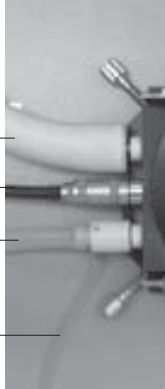
## Cooling Connections

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

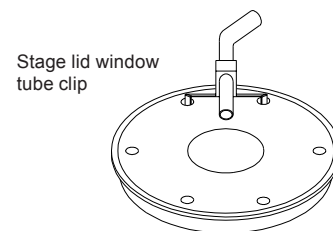
The thick white foam tubing is attached to the liquid nitrogen dewar. The thin black capillary tube inside the white foam tube must be inserted into one of the stainless steel cooling connectors on the stage. The white tubing slides on the outside of the stainless steel connector.

The thicker silicon tube from the LNP cooling pump ends in a white PTFE connector, this is pushed over the end of the other stainless steel connector as seen in the diagram opposite.

The smaller tube from the outlet marked WINDOW on the LNP should be placed in position on the top of the lid using the clip provided.



Dewar connector  
Stage connector  
LNP94 Pump connector  
Purging tube



### *Filling the Dewar*

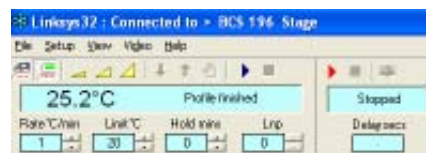
Fill the dewar 60mm from the top. The lid should be placed on before the liquid nitrogen stops boiling but do not fasten the clamps at this point.

When boiling has finished (noted by the decrease in bubbling noise), clamp the lid into place. This is a precaution to prevent the water vapour in the air condensing in the liquid nitrogen. If the lid is removed from the dewar flask, ensure that the filter is at room temperature before it is re-inserted. This will prevent any ice blockage. Care must be taken not to damage or kink the capillary tube as this will reduce the performance of the unit.

## Cooling Procedure

### Flushing the stage

1. Switch on the CI94 and set the limit to 40°C. Press the START button on the Linksys temperature control toolbar and wait until the limit is reached.
2. When connected to a DSC600 system, the LNP is set in the manual operation mode. Changing the pump speed is done by clicking on the 'increase' or 'decrease' pump speed buttons, seen on the toolbar above. Set this to the maximum pump speed.
3. Check that the valve opening insert is locked into the gas outlet valve on the right side of the stage. When pushing the insert into the valve, the outer valve jacket snaps forward to lock the insert.
4. Using a finger on your left hand, block the hole in the pump connector found on the opposite side to the purging tube. Still working with your left hand, pinch the narrow window tube to block it.
5. Continue blocking for 60 seconds, meanwhile regularly block and release the gas outlet valve with a finger on your right hand. 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 'O'rings in either the bottom or lid window.  
This pressurising procedure allows maximum mixing of the nitrogen with the residual air in the stage chamber so that the majority of the air is evacuated from the chamber.
6. With the block at 40°C any remaining water vapour will condense on the colder tubes carrying the coolant to the block.
7. Remove the valve opening insert by pressing the valve outer jacket toward the stage and unblock the pump connector and window tube.
8. After this initial set up, the temperature programmer can be set with the required limit and rate and used in the normal way. Adjust the LNP pumps speed according to the rate of cooling required. To conserve LN2 make this as low as possible. For DSC runs a cooling rate of 10C/min should only require fairly low pump speeds.



## Using the DSC

### *Preparing and Loading a Sample*

When working optically, to get a good reading from the DSC600 you need to use as much sample as possible, this is due to the sensitivity of the DSC element being impaired when using the sapphire crucible.

Running a calibration sample is only necessary if the actual sample shows excessive baseline drift. In most cases it will not be necessary to run a calibration.

If a calibration sample run is to be made, be sure to weigh the calibration sample using an accurate microbalance. You will have to weigh exactly the same amount of actual sample if you want to calibrate the actual sample curve.

The crucible is enclosed inside a silver chamber and sits on the DSC element within it.

Remove the lid of the silver chamber using a small cross head screw driver, the screws will not need much force to be undone and likewise require very little force when screwing back in.

Place the sapphire crucible inside the silver chamber making sure that the crucible does not touch the sides.



### *What happens during a run*

The programmer collects the difference between the PT 100 in the silver block and the DSC sensor.

If the signal of the sample's enthalpy is to be calculated it has to be computed from two runs. The first one is the calibration (cal) run, where the enthalpy of the crucible and any systematical contributions are collected. The second run is the sample data (dat) collection run, where the sample is in the crucible. By subtracting the cal run from the dat run, the DSC signal of the sample is obtained. The detection limit of an enthalpy was determined to be 0.16mg of Indium.

With the graph tools, the onset temperature and the peak area can be determined. By comparing the peak area of the sample with the peak area of a known substance, the enthalpy can be estimated. To give a good result, the reference material should melt in a temperature window of +/- 15°C of the sample's melting point.

Data is only collected from the DSC after ramp 1 is completed. This allows the user to raise the temperature quickly to the point of interest before beginning the actual run set by the values in ramp 2 and thereafter in the Data Table shown on page 13.

Data collection stops once the last ramp is complete or the Finish button is clicked.

## Data Capture Setup

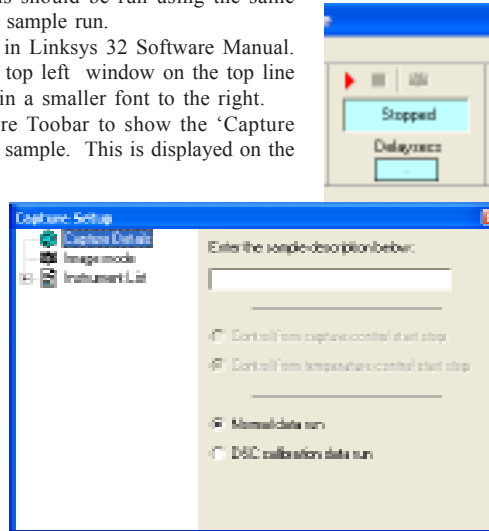
### *Calibration Run*

If a calibration run is required, a quantity of alumina should be prepared of the same weight as the actual sample. This should be run using the same ramp rates and limits as will be used in the sample run.

Start the Linksys 32 software as explained in Linksys 32 Software Manual. The temperature should be displayed in the top left window on the top line of the tool bar. The DSC output is shown in a smaller font to the right.

Right Click the mouse on the Data Capture Toolbar to show the 'Capture Setup' Window. Enter a description of your sample. This is displayed on the Data Capture Chart and on any captured images.

Click on 'DSC calibration data run' if intending to conduct calibration run.



Open the 'Temperature Profile Table' by clicking 'View > Temperature Profile'

Enter Rate, Limit and Time values into the table

DSC Data is not collected until the start of RAMP 2. Ramp 1 is simply used to bring the stage to the required starting temperature. This should have at least a 2 minute Hold Time so that the DSC has time to settle.

Click on the Start button in the programmer's toolbar. The stage will now begin heating or cooling depending on what has been programmed into the data table. Remember to select a suitable pump speed in the LNP pump speed window on the toolbar if you are cooling the sample.

During RAMP 2 and each ramp thereafter, data is collected from the DSC and will be written to the designated file. After the last ramp the programmer will switch off and a 'Save As' window will open. Type your designated file name.

### *Sample Run*

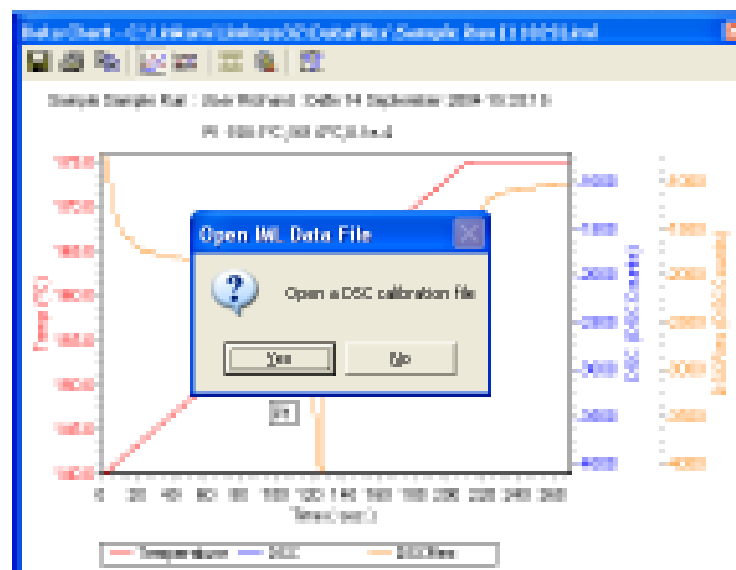
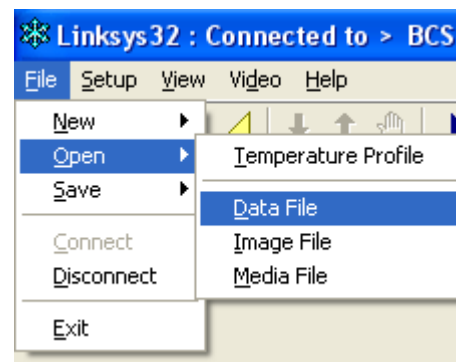
If you have done a Calibration run then weigh out exactly the same amount of sample, and load the crucible into the DSC as before.

As described above, open the 'capture setup' window and enter a sample description (The graph will use this description as a heading)

Select the 'Normal Data run' tick box. Use the same temperature profile as used in Calibration run. At the end of the run, a 'Save As' window will open. Type your designated file name.

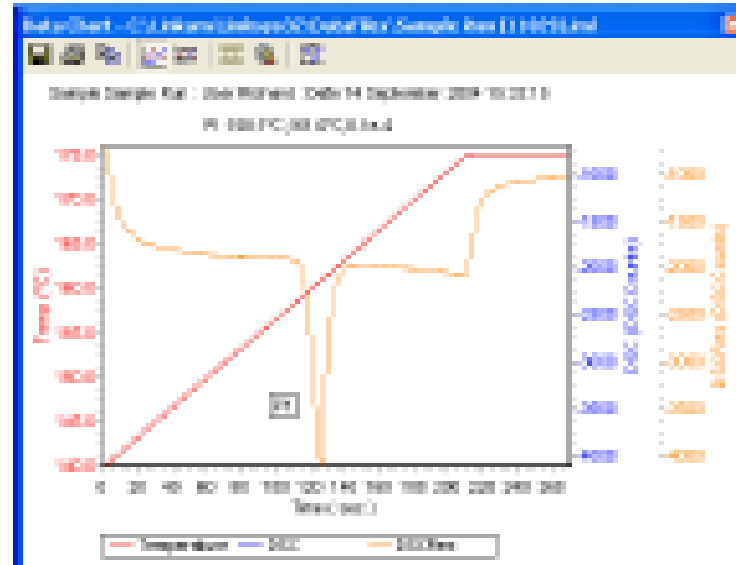
## Viewing the Data File

Go to 'Open > Data File

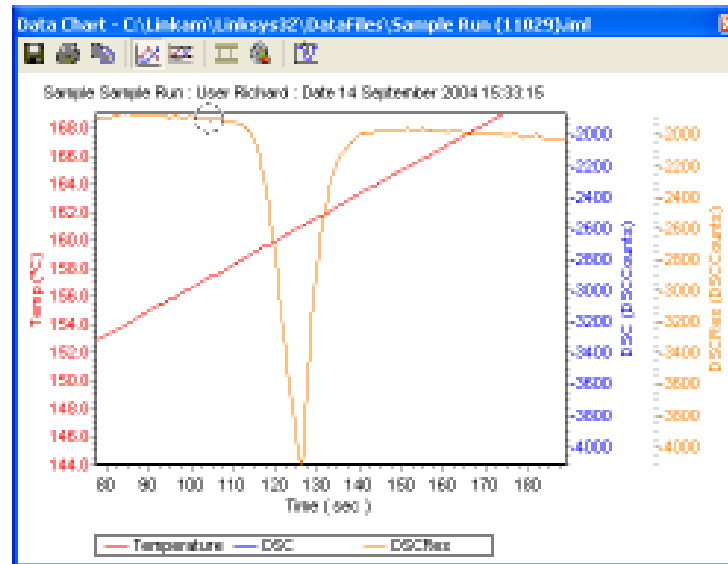



If no calibration file exists then click 'No'.  
The following images assume that 'No Calibration File' is being used.

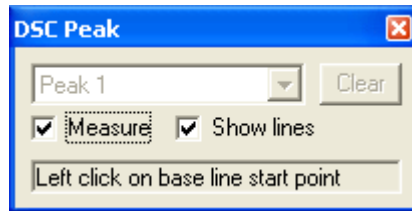




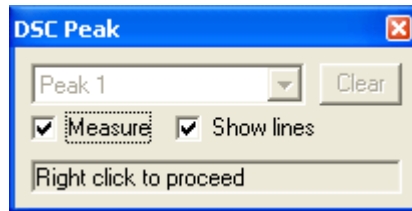
Magnify the Peak you are interested in by 'Right Clicking' the mouse and dragging a box around the peak.  
The image shows the magnified peak.



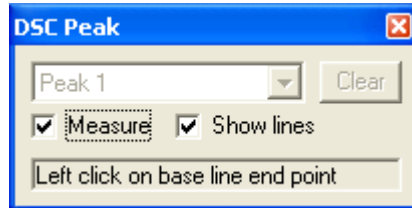
to measure the Peak, click on the DSC Peak Tool Button 



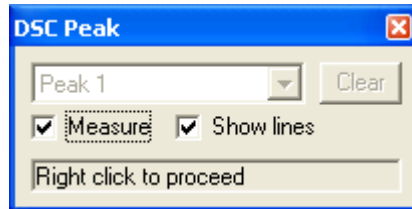
Move your mouse cursor onto the graph. A moveable round indicator line will show where on the line you are with your mouse cursor.  
You are asked to click on the base line start point. This will be the point where you want your base line to be taken from and can be just before the line starts to dip toward the peak.



Right click to move to the next measurement.

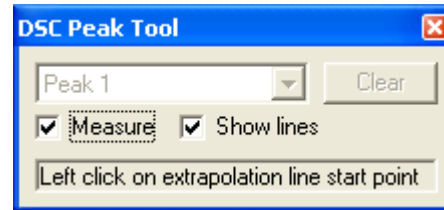


Now move your mouse cursor to the other side of the peak and click on the point where you believe the base line resumes, it is usually the point just after the peak line flattens out.



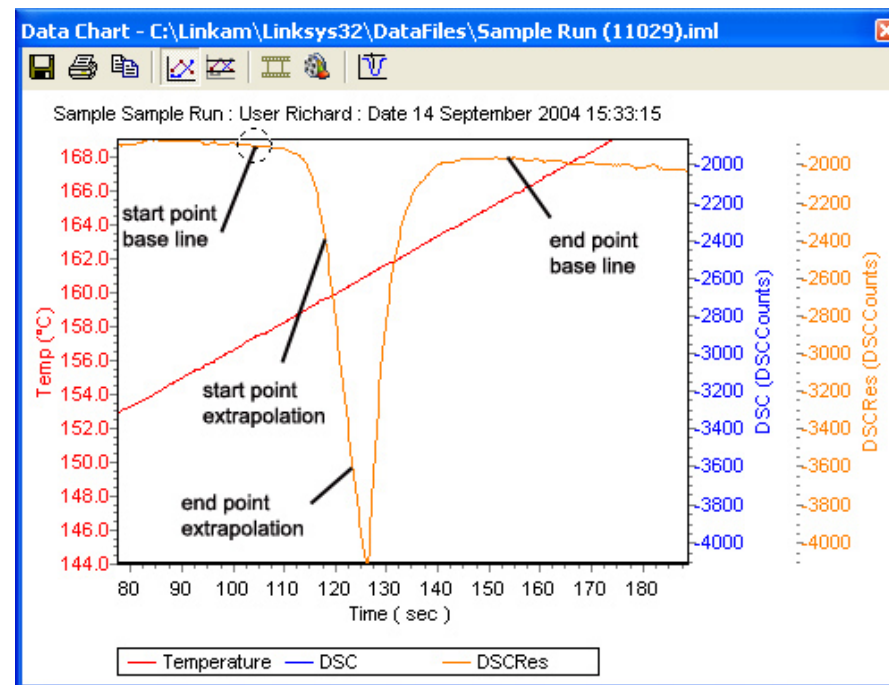
Right click to move to the next measurement.

You are now asked to click on the start of the extrapolation line. The extrapolation line is the vertical orientated line that is extrapolated so that it intersects with the hypothetical baseline. The start of the extrapolation line is the point at which the peak line is at it's steepest.

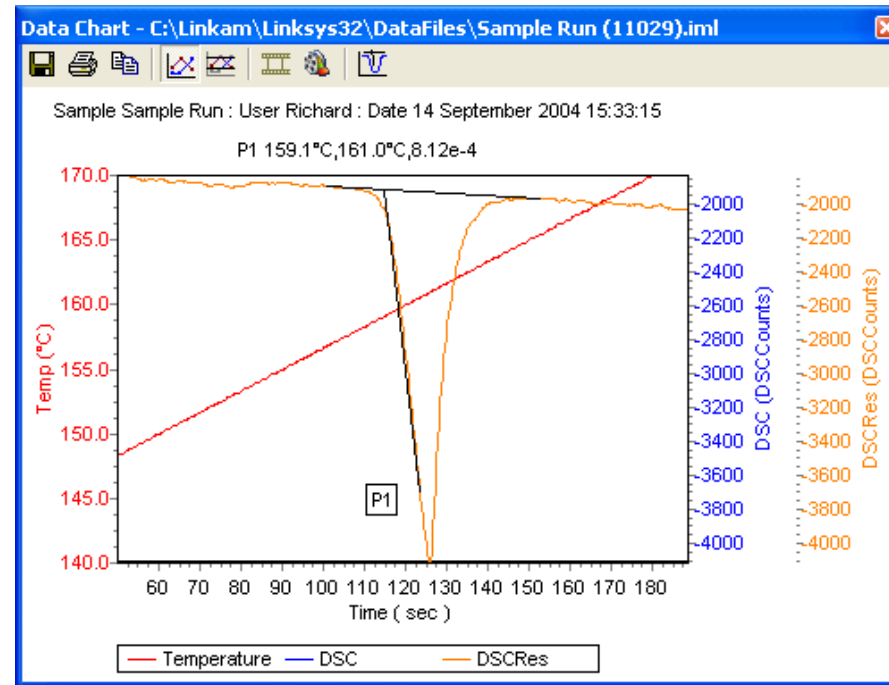


Right click to continue to move to next measurement

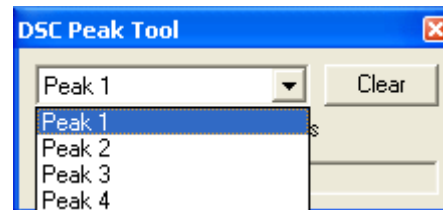
You are now asked to click on the 'End point of the extrapolation line.' This is a point further down the peak line that enables the software to make an extrapolation to the baseline.



The Peak measurements are shown at the top of the graph.

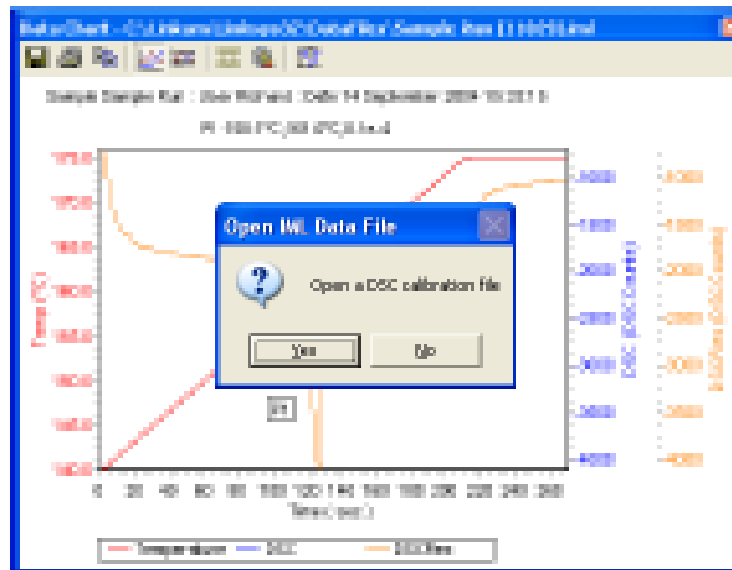


If there are more than one peak, you can now select the next peak from the drop down menu.



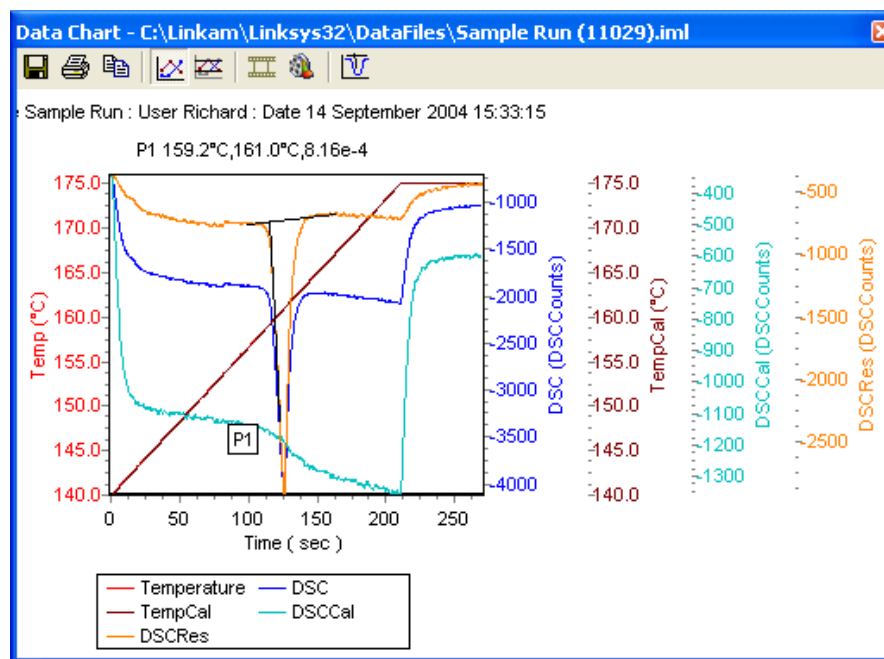
By moving your mouse cursor along the graph trace for DSC counts or temperature, the measurement at that point will be shown in a small yellow box.

Using a Calibration file



Click on 'Yes' and open the Calibration file you want to use.





#### Data Chart Description

There are five different vertical scales on this graph. The first scale on the left side of the graph is the temperature scale of the sample run and is shown in red. The temperature graph line running diagonally from the bottom left to top right is also in red but cannot be seen very well as it is overlaid by the Temperature Calibration run (Purple colour-Temp Cal(°C)). **Note: Cal run must use same temperature profile as sample run, hence both Temperature Cal and Temperature Sample lines follow same path on the graph. (e.g 10°C/min)**

The next scale on the right of the graph is DSC counts of the sample run and is shown in blue.

The next scale on the right is the graph of Temperature Profile of the Calibration run and is shown in purple (overlays Temperature Profile of Sample run).

The next scale on the right is the DSC counts of the Calibration run (e.g empty crucible) and is shown in turquoise.

The next scale on the right is the DSC resultant line reached by combination of sample run and calibration run and is shown in red.

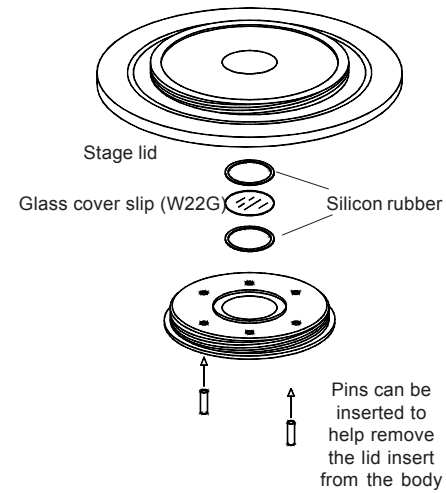
Use this line to make the peak measurement (described on previous page).

## Fitting the Windows

### *DSC Lid Assembly*

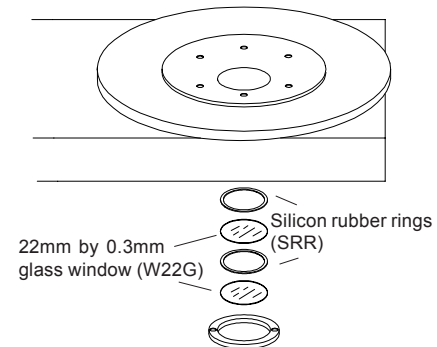
To replace the windows in the lid, the lid and insert should first be unscrewed from the body and taken apart.

The Stage lid insert should be turned upside down as shown and reassembled in the order indicated. The Stage lid should be screwed down until the cover slips are held firmly, then turn the assembly over and screw down the insert until it is felt to come to a stop.



### *DSC Bottom Window Assembly*

If for some reason the bottom windows need to be replaced they should be reassembled as shown in the diagram.



### *LNP94/2 Fault Diagnosis*

Ensure that all connections to the stage and dewar are as shown in the diagram and that the stage lid and top windows are properly sealed.

1. *The cooling rate is less than 30°C/minute.*

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. This is particularly important on the stage connector as any constrictions of either the tubing or the connector will have a drastic effect on the cooling ability of the LNP94

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 coolant pipes.

2. *Stage will not cool down to -194 °C or beyond -150C.*





Check that the stage lid is not touching the silver block when screwed down as this will cause a substantial loss of cooling ability.

3. *Condensation and ice forming on the window.*

Realign the window gas tube clip to the required position in the stage lid.



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