

STAGE REFERENCE MANUAL



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Issue 2: 21-04-97

OPTICAL INFORMATION

It cannot be stressed enough, how important it is to have the microscope set up correctly, especially when working at high magnifications or when looking at difficult objects such as small fluid inclusions. 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 setting up procedures for the microscope, a simple line drawing showing the essential parts of the microscope's optical path is shown opposite.

If the condenser working distance is less than 12.5 mm, we can supply a condenser extension lens for both the Olympus and Nikon microscopes under part numbers CELO and CELN respectively (see drawing). For other microscopes please contact us.

NOTE:-If the Stage clamps are used with the Olympus microscope the CELO lens is NOT required.

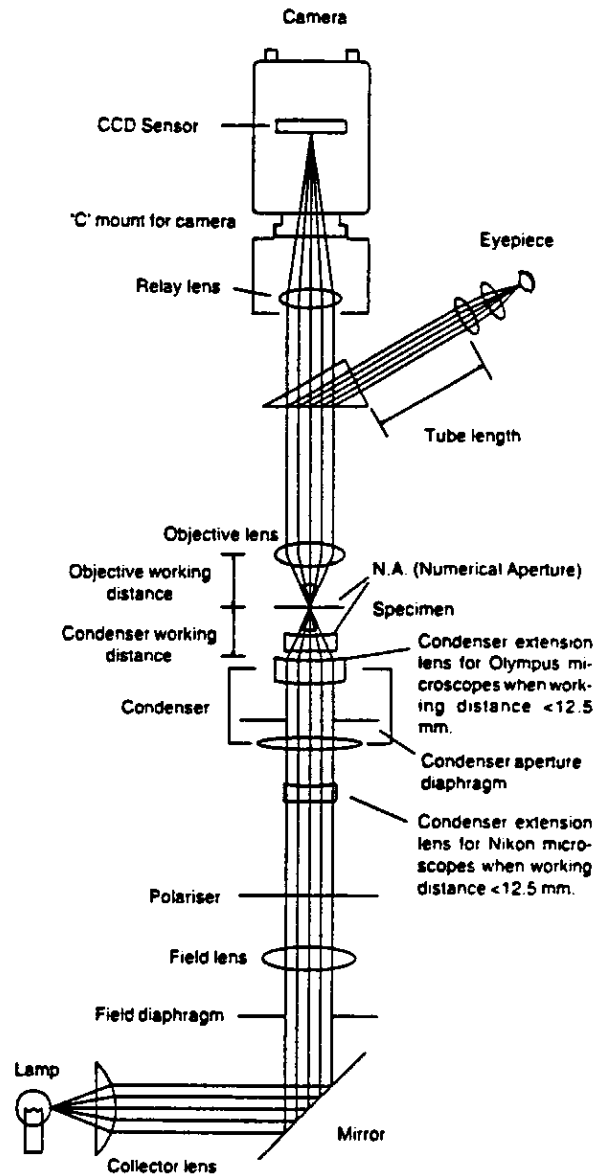
Phase Contrast

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

Light source centring and focus

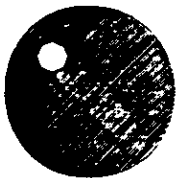
The adjustment of the light source is only necessary when the microscope is first installed or when the bulb is replaced, provided that the adjustment's made are not disturbed.

- 1) Open the field diaphragm on the base of the microscope to it's 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.



Centring the condenser lens

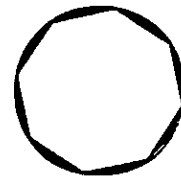
It is extremely important to have a focused, centred and condensed light beam at all times and to ensure that the field diaphragm and condenser aperture diaphragm are set correctly.



1) Select the 10X lens and close the field diaphragm on the microscope base to it's 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

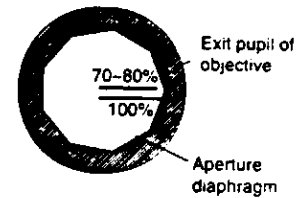
The field diaphragm controls the diameter of the illuminated area on the specimen surface in relation to the field of view of the microscope. By stopping down the field diaphragm until it is slightly larger than the field of view, it can reduce stray light, which in turn increases image definition and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

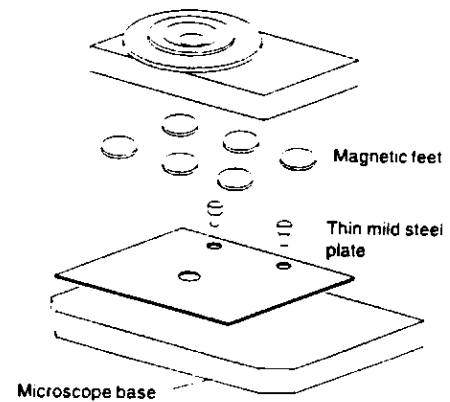
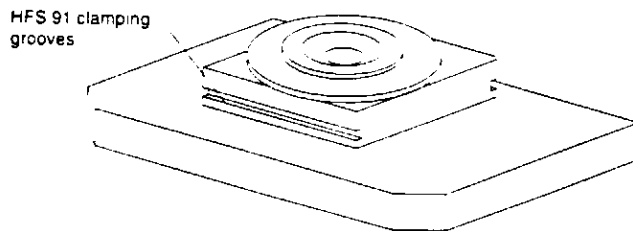
In general it should be stopped down to 70-80% of the numerical aperture of the objective lens for best overall results.

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.



MICROSCOPE MOUNTING

Six self adhesive magnetic feet are supplied which can be used together with a thin mild steel plate to attach the hot stage to the microscope's mechanical base. The HFS91 can also be mounted using the clamping grooves on the side of the body. If it is not practical to use the magnetic feet, double-sided adhesive tape can be used.



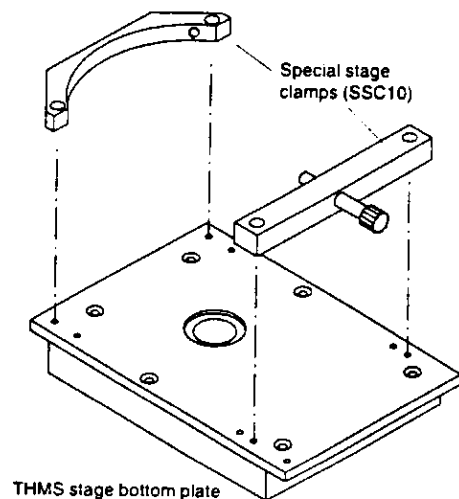
For very high magnification and stability, especially when using the LNP cooling unit, the microscope's X,Y table can be removed and the THMS stage fitted directly to the microscope's circular sub-Stage.

MOUNTING ARRANGEMENT FOR NIKON AND OLYMPUS MICROSCOPES

This arrangement uses a set of SSC10 stage clamps which are simply screwed to the bottom of the existing THMS body using four cap screws which are provided. Some of the older THMS stages will not have the correctly drilled bottom plate and will have to be modified. Contact us for details.

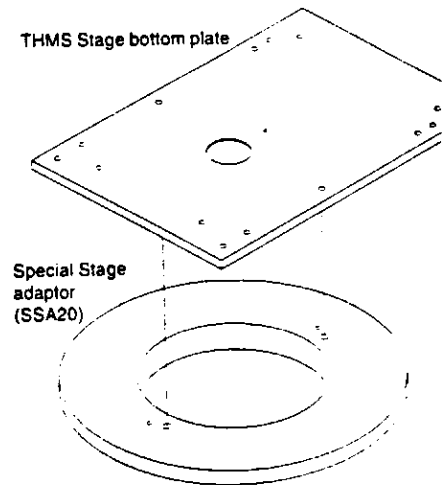
The three positioning screws are used to centre the silver block window as follows.

- 1) Loosen the three screws and position the stage and clamps on the microscope's sub-stage.
- 2) Select a 10X lens and focus on the window.
- 3) Using the long screw, adjust the position of the stage in the X direction until it is centred.
- 4) Gradually adjust and tighten the two smaller screws in order to adjust the stage in the Z direction.



MOUNTING ARRANGEMENT FOR ZEISS AXIOPLAN AND AXIOPHOT MICROSCOPES

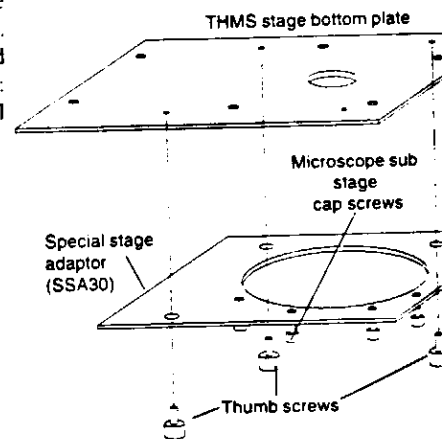
For the Zeiss Axioplan and Axiophot Microscope, a special adaptor is available which is screwed to the bottom of the THMS body using the two 12mm screws provided. These replace the two 6mm screws already used in the base plate. In order to centre the heater block and window, adjust the microscope's spring loaded screws which hold the SSA20 in place. If this is not enough adjustment, the SSA20 adaptor can be moved slightly by loosening the mounting screws and moving the stage body before re-tightening them again.



MOUNTING ARRANGEMENT FOR ZEISS AXIOSKOP MICROSCOPES

For the Zeiss Axioskop a special adaptor is available which is screwed to the bottom of the THMS body using the three thumb screws provided (see diagram). As these screws are also used for centring the heater block and window they should not be tightened until the complete assembly is on the sub-stage. Fit as follows:

- 1) Screw the SSA30 to the THMS body using the screws provided until they are finger tight.
- 2) Mount the complete assembly to the Zeiss sub-stage using the four screws already in the Zeiss sub-stage.
- 3) Select a 10X lens and centre the heater block and window by moving the THMS body. Tighten the mounting screws.



OBJECTIVE LENSES

For use at high magnification special lenses are required which have long working distances, a selection of which are shown below. Some of the Olympus lenses may be used on the Nikon Microscope but because of tube length differences the magnification may not be exact. This is also the case if Nikon lenses are used on Leitz Microscopes.

For correct lens magnification the lens must be matched to the tube length of the microscope (see optical drawing).

A list of lenses suitable for use on the heating/freezing stage is detailed below :

Objective Lens	Numerical Aperture	Working Distance	Special Lid	Tube Length (mm)
Olympus				
Achromat 10X	0.25	6.1		160
Plan 10X	0.25	10.5		160
Plan 10X Phase Contrast	0.25	10.5		160
Ultra long working distance MS plan Achromat 80X	0.75	4.1	Yes	∞
LM Plan F1 20X		12		∞
LM Plan F1 50X	0.5	12		∞
LM Plan F1 100X		3.4		∞

Leica

559121 L10/0.22P Long working distance 10 times polarised	0.22	15.8		160
559122 L20/0.32P Long working distance 20 times polarised	0.32	6.73		160
559123 L25/0.22P Long working distance 25 times polarised	0.22	14.7		160
559124 L32/0.40P Long working distance 32 times polarised	0.40	6.55		160
519760 EF10/0.25 Long working distance 10 times	0.25	6.78		160
519434 EFL20/0.32 Long working distance 20 times	0.32	6.73		160
519679 LL25/0.35 Long working distance 25 times	0.35	13.7-14.3		160
519435 L32/0.40 Long working distance 32 times	0.40	6.55		160
518087 EF10X Phase Contrast 1A	0.25	6.78		160
518076 EFL20X Phase Contrast 1A	0.32	6.73		160
518077 L32X Phase Contrast 1A	0.40	6.55		160
569231 NPL FL 10/0.22 Infinity corrected	0.22	11.8		∞
569216 LL 20/0.40 Infinity corrected	0.40	10.1		∞
569244 PLAN L25/0.40 Infinity corrected	0.40	11.0		∞
569247 PLAN L50/0.60 Infinity corrected	0.60	6.8		∞
569107 H 20/0.40. For high temp TH1500. Corrected for 1.8 mm quartz.	0.40	8.2		∞
569109 H 32/0.60. For high temp TH1500. Corrected for 1.8 mm quartz.	0.60	5.8		∞
567002 PLANH 20/0.40. For high temp TH1500. Corrected for 1.8 mm quartz.	0.40	12.6		∞
567003 PLANH 40/0.60. For high temp TH1500. Corrected for 1.8 mm quartz.	0.60	7.1		∞
567000 PLAN L20X.		11.0		∞
567004 PLAN L40X.		6.8		∞

Nikon

MSK 00102 CF E PLN LWD 10X (cover glass corrected)	0.25	10.50		160
MSJ 05200 CF N PLN ELWD 20X (cover glass corrected)	0.40	6.00		160
MSJ 05400 CF N PLN ACH ELWD 40X (cover glass corrected).	0.55	5.08-6.84		160
Recommended for stage use				
MTJ 60100 CF M PLN ACH 10X	0.25			210
MTJ 63200 CF ELWD M PLN ACH 20X				210
MTJ 63400 CF ELWD M PLN ACH 40X	0.5	10.1		210
MTJ 63600 CF ELWD M PLN ACH 60X		4.90	Yes	210
MTJ 67900 CF SLWD M PLN ACH 100X	0.75	4.7	Yes	210

Nikon phase contrast objectives

The entire Nikon range of phase contrast objectives may be used with the Linkam THMS 600 series stages without the stage lid insert in position. The long working distance phase contrast objectives must be used when the THMS 600 stage is used with the stage lid insert in position. (Please consult Linkam Scientific Instruments Ltd.)

MSJ10041 CF N PLN ACH DL 4X	0.13	16.22		160
MSJ10101 CF N PLN ACH DL 10X	0.30	9.22		160
MSB10101 CF A CH DL 10X	0.25	5.60		160
MSK10101 CF E ACH DL 10X	0.25	5.20		160
MSJ13200 CF N PLN ACH ELWD DL 20X	0.40	6.00		160
MSB13400 CF N PLN ACH ELWD DL40XC (Corrects glass 0-2.5 mm thick)	0.55	5.08-6.84		160

Objective Lens	Numerical Aperture	Working Distance	Special Lid	Tube Length (mm)
Carl Zeiss				
440120 Non flat Achrostigmat 5X (cover glass corrected)	0.12	11.0		∞
440130 Non flat Achrostigmat 10X (cover glass corrected)	0.25	6.5		∞
440020 Flatfield Achroplan 4X (cover glass corrected)	0.1	11.5		∞
440320 Flatfield Plan Neofluar5X (cover glass corrected)	0.15	13.6		∞
440330 Flatfield Plan Neofluar10X (cover glass corrected)	0.30	5.6		∞
440844 Flatfield Achroplan LD20X (cover glass corrected)	0.4	10.9		∞
440850 Flatfield Achroplan LD32X (cover glass corrected)	0.4	6.9		∞
442840 Flatfield Epiplan LD 20X	0.4			∞
44285 Flatfield Epiplan LD 20X	0.5	7.0		∞

CONDENSER LENSES

Leica

513501	0.7	4.0
513502	0.55	15
513538	0.35	30

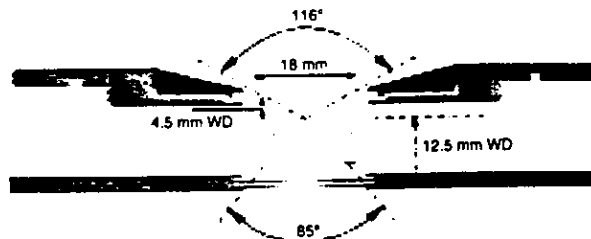
CONDENSER EXTENSION LENSES

Olympus

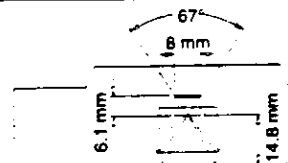
Microscope Condenser Type Working Distance Linkam Part No	BX Phase Contrast U-PCD 14mm OLW-UPCD	BX Long Working Distance U-LWCD 14mm CELO	BX Swing Out Lens U-SC 14mm OLW-U-SC	BX Universal Condenser U-UCDB 3mm OLW-UCDB	BX Universal Condens U-UCDB 14mm OLW-UCDB
Lens Appearance					
Notes	Replaces existing lens assembly. Use phase number two for 10x and 20x lenses. Use the Linkam phase ring for a 40x lens.	Place lens on long working distance condenser if the microscope table is to be used.	Replaces the top condenser lens	Remove the microscope's top swing out lens assembly. The Linkam condenser is built inside the BCS and MDS stages.	Unscrew the condenser and replace with the Link Suitable for phase cont DIC. Use phase number and 20x lenses Use the phase ring for a 40x lens
Linkam Phase Ring	OLPR-UPCD				OLPR-UCDB

Nikon

Optiphot/Eclipse Phase Contrast 0.9 Dry 14mm NK-PCD	Optiphot/Eclipse LWD Achromat Condenser 13mm CELN	Optiphot/Eclipse Achromat Swing Out Condenser 14mm NK-SOC	Optiphot/Eclipse Phase Contrast Turret Condenser -2 3mm NK-PCTC	Optiphot/Eclipse Universal Condenser NK-UC
Replaces existing lens assembly For 10 and 20x lenses remove ring PH1 in the condenser and replace it with the Linkam Phase Ring NK-PH1. For the 40x lens remove ring PH2 in the condenser and replace it with the Linkam Phase Ring NK-PH2.	Place adaptor in the bottom of the LWD Achromat condenser.	Replaces top lens	Remove the microscope's lens assembly and replace with the adaptor ring NK-PCTC. The Linkam lens is built inside the BCS and MDS stages.	Under development. Please contact Linkam for the latest details.
NK-PH1 NK-PH2				

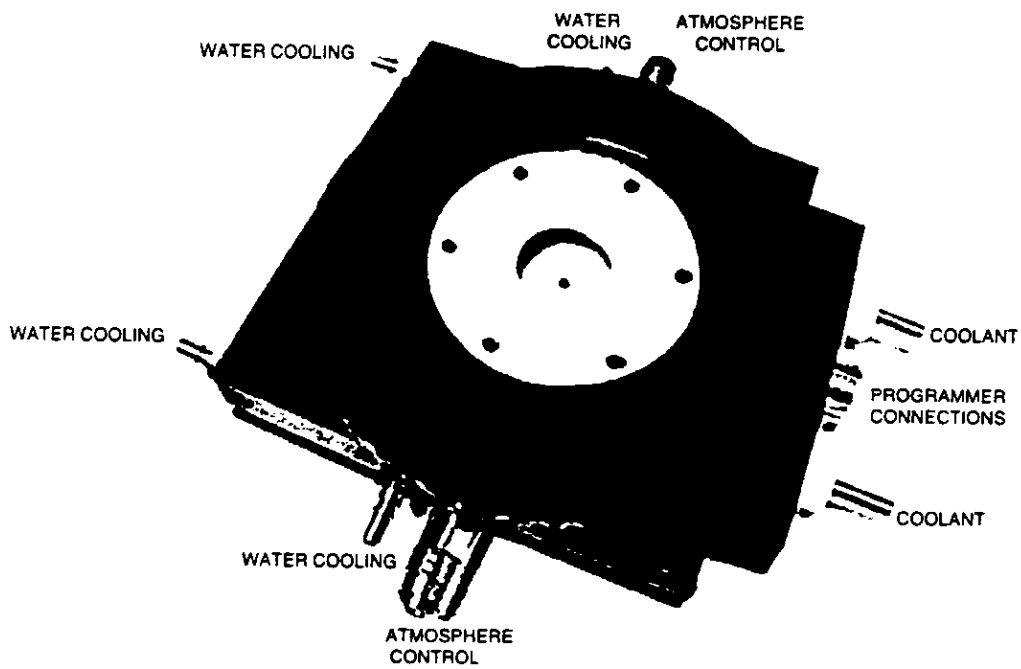
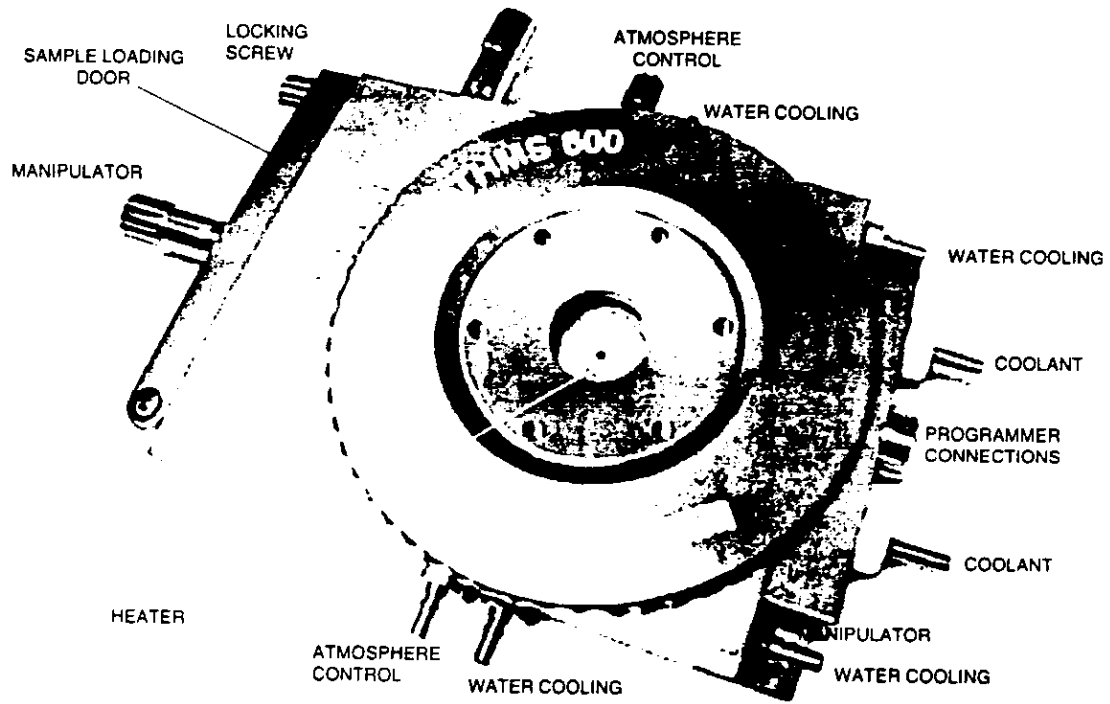


Angles and working distances of the THMS and HFS stages fitted with an FTIR silver block



57°
TS 1500

THMS 600 / HFS 91 STAGE CONNECTIONS



THMS600 SAMPLE PREPARATION AND ACCESSORIES

For accurate results it is most important that the surface of the block, window and carrier are extremely clean as any particles of dust, grease or skin will cause air gaps between the block and the window resulting in temperature errors. The block is made of silver and being so soft it can get scratched when a sample or cover glass is placed upon it, especially by using sharp fine tweezers. This could cause a ring preventing the cover slip from sitting flat on the block. The resulting air gap would then cause temperature errors.

For this reason we recommend that the sample and windows are always handled using the Linkam vacuum tweezers.

There are a number of different ways in which the sample may be positioned on the silver block depending on the application and the type of sample carrier.

Single cover slip and crucible carrier

The simplest arrangement is shown in Fig 1 where one of the stainless steel rings is inserted through the crucible carrier onto the block and the cover slip and sample are then inserted inside the ring. The crucible carrier then moves the ring and cover slip around the block. To place the assembly, the stage lid must be removed.

This is adequate for many applications but for precision work the assembly shown in Fig 2 should be used.

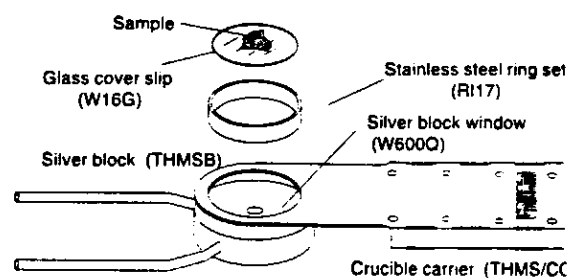


Fig 1

Multi-ring assembly with crucible carrier

The assembly of cover slips and stainless steel rings shown in Fig 2 form a 'double glazed' environment around the sample. This traps a layer of air above the sample and avoids draughts and air turbulence from affecting the sample temperature hence giving more accurate results. The assembly of rings and cover slips are placed on the silver block by removing the stage lid. If used with a THMS600, the rings may be moved around the silver block using the crucible carrier.

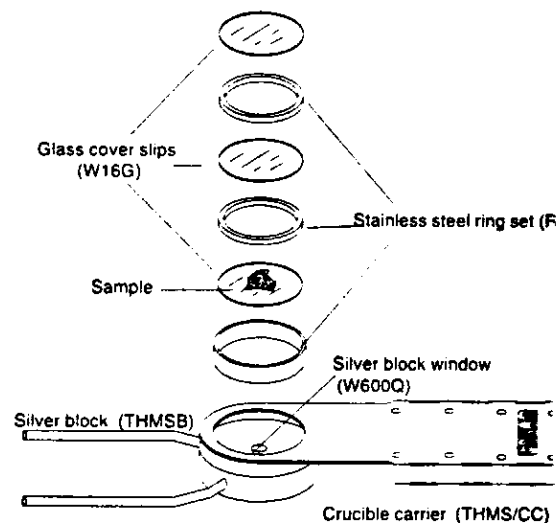


Fig 2

Quartz Crucible

The advantages of using a crucible are that it can be used to hold liquids and can load a sample through the side loading door of the stage without removing the lid or disturbing the lens set up.

The crucible is made from fused quartz and has a small lip around the top which allows it to sit inside the crucible carrier without falling out. For better temperature accuracies the sample should be covered with either a 16 mm glass cover slip over the top of the crucible or by a 13 mm glass cover slip over the sample inside the crucible.

When using the crucible check that the lid does not touch the top of the assembly. To use this fixed window crucible the THMS 600 must be fitted with a guide ramp.

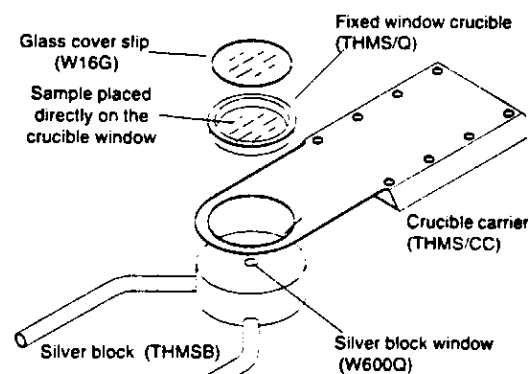


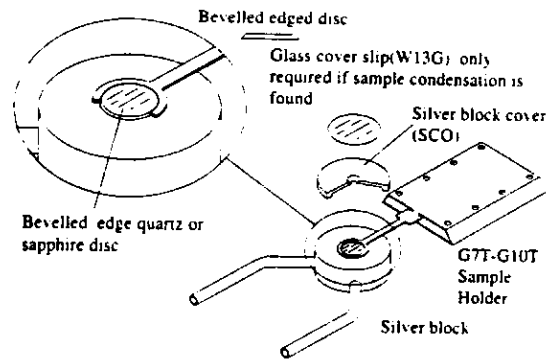
Fig 3

G7 and G10 Sample Holders with Silver Block Cover

These sample holders are designed to take the 7 or 10mm quartz discs. These discs are supplied with a bevelled edge as shown opposite and are 'pushed' down onto the block using the spring action of the sample holder. The silver block cover is placed over the sample and its holder to form a chamber with a very even temperature gradient.

The silver cover has a small slot in the side to allow the sample holder to move the cover slip around the surface of the block. A cover slip can be placed over the silver cover if sample condensation is noticed.

The cover slip and sample must be loaded when the cover is removed and cannot be used with the side door of the stage. To load the sample the carriers are pushed into place slightly overlapping the lollipop holder onto the silver block. The wider edge of the bevelled disc is placed on the block next to the head of the 'lollipop', by slightly bending the lollipop holder up over the disc and simultaneously moving the holder further into the block the bevelled



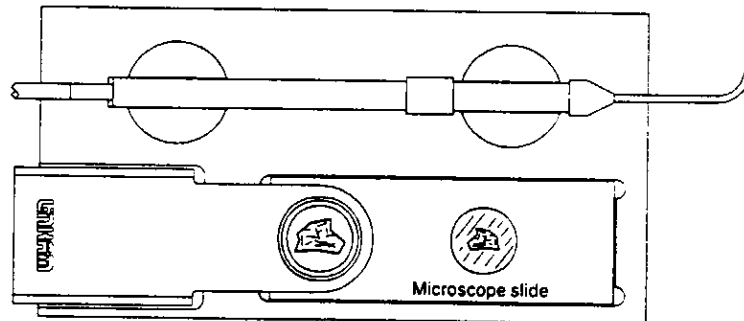
edge of the holder fits into the bevelled edge of the disc, the spring action of the holder then holds the disc securely in place on the block.

THMS600 SPARES

CRUCIBLE CARRIER	THMS/CC
QUARTZ CRUCIBLE	THMS/Q
SET OF STAINLESS STEEL RINGS	RI17
16 mm BY 0.2 mm GLASS COVER SLIPS (100 OF)	W16G
2.4mm BY 1.6mm QUARTZ WINDOW FOR SILVER BLOCK	W600Q
13 mm BY 0.2 mm GLASS COVER SLIPS (100 OF) FOR USE IN CRUCIBLE	W13G
SILVER BLOCK COVER	SCO
7 mm Tapered SAMPLE CARRIER FOR USE WITH SILVER BLOCK COVER	G7T
10 mm Tapered SAMPLE CARRIER FOR USE WITH SILVER BLOCK COVER	G10T
7 mm Quartz tapered window	W7QT
10 mm Quartz tapered window	W10QT

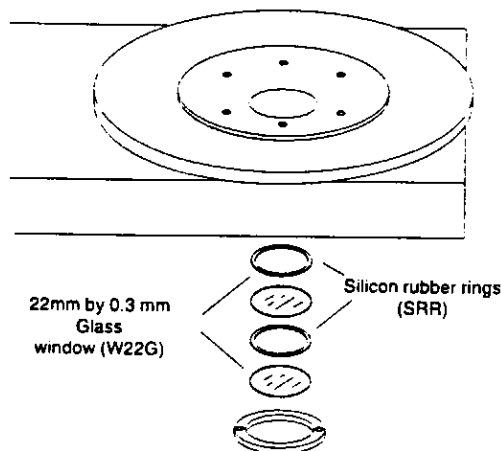
Sample Loading Station

The sample loading station provides a clean and easy way to store the vacuum tweezers, crucible carrier and a microscope slide. Additional windows and samples may be kept on the station ready for loading into the carrier. Placing the window is also easier as the carrier is held in a flat position. As the station holds the carrier flat, it makes the insertion and positioning of a new window using the vacuum tweezers much easier.



THMS600/HFS91 BOTTOM WINDOW ASSEMBLY

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



HFS91 SAMPLE PREPARATION

For accurate results it is most important that the surface of the block, window and carrier are extremely clean as any particles of dust, grease or skin will cause air gaps between the block and the window resulting in temperature errors. The block is made of silver and being so soft it can get scratched when a sample or cover glass is placed upon it, especially by using sharp fine tweezers. This could cause a rise preventing the cover slip from sitting flat on the block. The resulting air gap would then cause temperature errors. For this reason we recommend that the sample and windows are always handled using the Linkam vacuum tweezers. There are a number of different ways in which the sample may be positioned on the silver block depending on the application and the type of sample carrier.

Single cover slip

The simplest arrangement is shown in Fig 1 where the sample is placed upon a cover slip and which is then mounted on top of the silver block. To place the assembly the stage lid must be removed. This is adequate for many applications but for precision work the assembly shown in Fig 2 should be used.

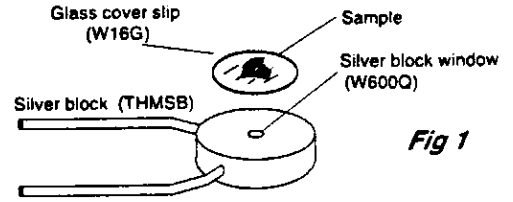


Fig 1

Multi-ring assembly

The assembly of cover slips and stainless steel rings shown in Fig 2 form a 'double glazed' environment around the sample. This traps a layer of air above the sample and avoids draughts and air turbulence from affecting the sample temperature hence giving more accurate results. The assembly of rings and cover slips are placed on the silver block by removing the stage lid.

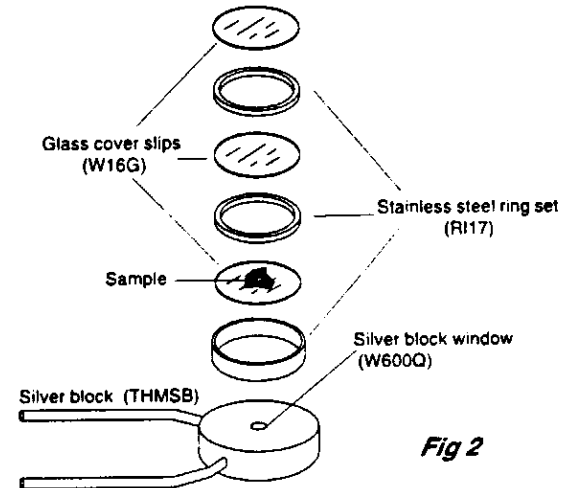


Fig 2

Quartz Crucible

The advantages of using the crucible are that it can be used to hold liquids and to load a sample through the side loading door of the stage without removing the lid or disturbing the lens set up.

The crucible is made from fused quartz and has a small lip around the top which allows it to sit inside the crucible carrier without falling out.

For better temperature accuracies the sample should be covered with either a 16 mm glass cover slip over the top of the crucible or by a 13 mm glass cover slip over the sample inside the crucible.

When using the crucible check that the lid does not touch the top of the assembly.

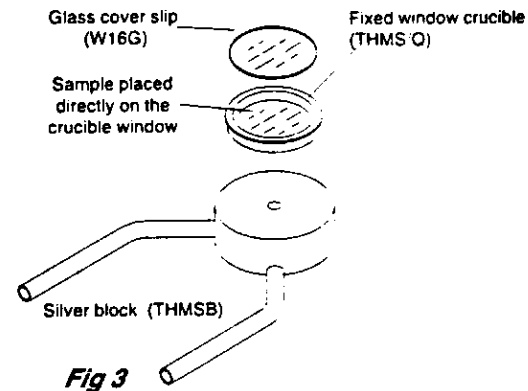
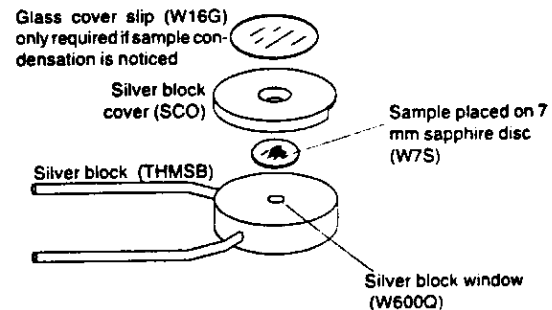


Fig 3

Silver Block Cover

An additional option which can be used to improve the temperature gradient across the sample even further is to place a cover over the silver block to form a miniature 'furnace'. The arrangement shown here uses only one glass cover slip over the silver cover and one in the top lid assembly as shown on the next page. However, if there is no condensation on the sample, the cover slip over the silver cover can be removed.



HFS91 SPARES

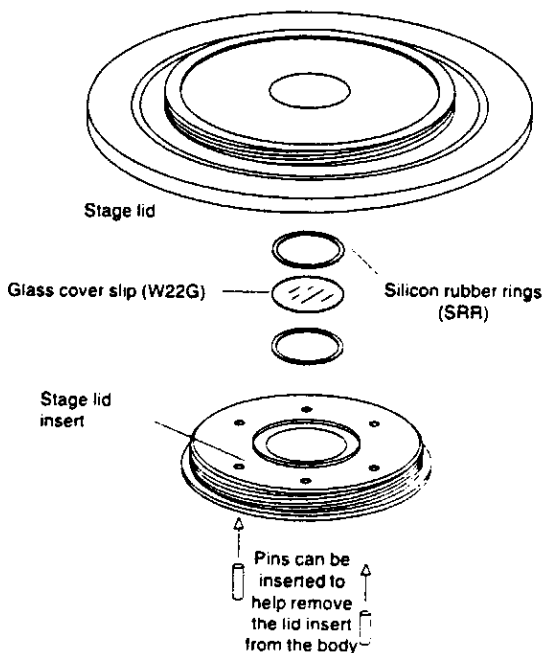
QUARTZ CRUCIBLE	THMS/Q
SET OF STAINLESS STEEL RINGS	RJ17
16 mm BY 0.2 mm GLASS COVER SLIPS (100 OF)	W16G
2.4mm BY 1.6mm QUARTZ WINDOW FOR SILVER BLOCK	W600Q
13 mm BY 0.2 mm GLASS COVER SLIPS (100 OF) FOR USE IN CRUCIBLE	W13G
SILVER BLOCK COVER	SCO
7 mm Quartz tapered window	W7QT
10 mm Quartz tapered window	W10QT

THMS600/HFS91 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 re-assembled 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.



THMS600 / HFS91 LID SPARES

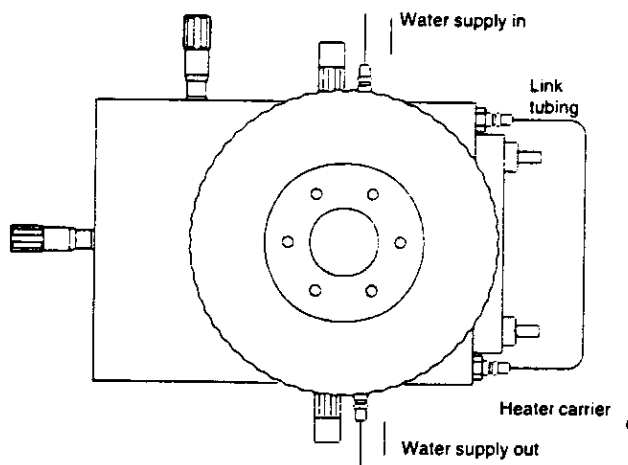
22 mm BY 0.2 mm GLASS COVER SLIPS (100 OF)	W22G
22mm BY 0.5 mm QUARTZ WINDOW FOR TOP AND BOTTOM STAGE WINDOWS	W22Q
SILICON RUBBER RINGS	SRR
22 mm by 0.3 mm Glass cover slips for bottom window and for vacuum top lid	W22G0.3

USING THE THMS600 / HFS91 STAGE

A length of silicon tubing is supplied which should be used for the top window. (i.e. to pass dry nitrogen through, this prevents condensation of the window at low temperatures) and for liquid nitrogen when used to connect to the copper cooling coils of a dewar flask.

Also supplied are four snap-on connectors for use with 3mm inside diameter, 6mm outside diameter flexible tubing. These are used for connecting both gas and water to the main body of the stage, although water cooling is only required if the stage is to be used at temperatures greater than 300°C and periods longer than fifteen minutes.

Either continuous mains water or a water circulator can be used and should be connected as shown in the diagram. A circulator is available from Linkam and should be used as detailed on Page 18. The HFS 91 should be connected in the same way i.e. a small length of tubing between the two connectors on the heater carrier.



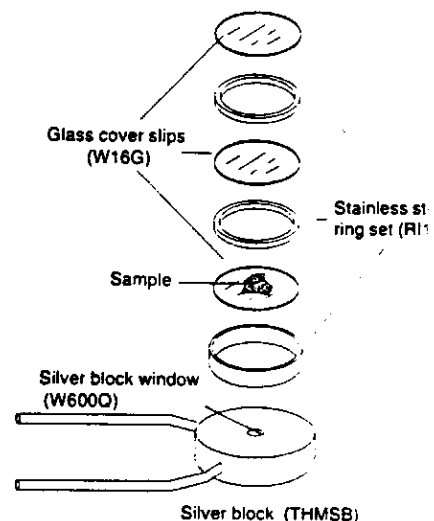
THMS600 / HFS91 SPARES

Platinum resistor 1.6 mm dia by 15 mm, 100 ohms PT100	
Set of O ring seals for the stage body and lid	ORTHMS
Plug for electrical connection on the THMSE or HFSE	LE2
Silver block heater assembly (exchange only)	THMSB

SAMPLE CALIBRATION FOR THMS600/HFS91 STAGES

The stages have been carefully designed to minimise any temperature difference between the heater and the sample. This has been achieved by enclosing both the platinum resistor and the heater within a high thermal conductivity silver block. The sample itself can be placed directly upon a cover slip or can be used in the 'double glazed' environment shown in the diagram. This arrangement of cover slips and stainless steel rings traps a layer of air above the sample thus avoiding any draughts and turbulence.

With thicker materials there may be a temperature difference between the sample and the silver block which gives an incorrect temperature reading on the programmer when the sample melts. By using the sample calibration feature of the programmer this error can be eliminated. A sample of known melting point is used to set an end point of a straight line which must pass through zero. An end point temperature can be set for both a positive and a negative value. The zero value can also be determined and entered. These values are entered as six temperatures in the following order: actual negative value, measured negative value, actual zero value, measured zero value, actual positive value and finally the measured positive value. Details of how to enter these values can be found in the programmer's manual under SAMPLE CALIBRATION FOR THE THMS600/HFS91 STAGES.



Sample calibration substances

THE FOLLOWING SUBSTANCES AND THEIR MELTING POINTS ARE GIVEN AS A REFERENCE ONLY AND MAY BE DANGEROUS IF HANDLED INCORRECTLY

The melting point values for Benzoic Acid and 2-Chloroanthraquinone are mean values taken from the dynamic and static methods by the National Physical Laboratory.

SUBSTANCE	SYMBOL	T °C	MELT DESCRIPTION
Toluene	$C_6H_5CH_3$	-95	Simple digestion of needle by fluid which forms at the grain boundaries and within the needles.
Chloroform	$CHCl_3$	-63.5	Droplets of fluid appear within the crystals and progressively form a melt at the expense of a solid.
Synthetic Fluid Inclusion	CO_2	-56.6	
N-Octane	$CH_3(CH_2)_6CH_3$	-56.5	Droplets of liquid which can form internally and digest the small blades.
Chlorobenzene	C_6H_5Cl	-45	Droplets of liquid which form within the large blades and digest them.
Carbon Tetrachloride	CCl_4	-22.8	This coarsely crystalline aggregate changes to a featureless solid at -33°C. Droplets of melt form within the solid and digest it.
N-Dodecane	$CH_3(CH_2)_{10}CH_3$	-9.6	A melt forms at the blade boundaries and the solid gas-bubble contact points.
Water	H_2O	0	Forms a coarsely crystalline aggregate on freezing. The melting occurs at the grain boundaries which appear more prominent before fusion.
Benzoic Acid	C_6H_5COOH	122	
2-Chloroanthraquinone	$C_{14}H_8O_2Cl$	210	
Potassium Nitrate	KNO_3	333	Phase change occurs at 127°C.
Potassium Dichromate	$K_2Cr_2O_7$	398	

Standard Accessories supplied with THMS 600

1x	PVC Tube
2x	CONHV Water hose connectors
2x	CONHS Gas connections
6x	Magnetic feet for stage mounting
4x	Silicone washers
1x	RI 17 Stainless steel ring set for sample preparation
1x	TCH Tube clip holder for stage lid
1x	Crucible carrier (THMS/CC)
50x	16mm glass cover slips for sample preparation (W16G)
50x	22mm glass cover slips for stage windows (W22G)
10x	22mm x 0.3mm glass cover slips (W22G0.3)

Standard Accessories supplied with HFS 91

1x	PVC tube
2x	CONHV Water hose connectors
2x	CONHS Gas connections
6x	Magnetic feet for stage mounting
4x	Silicone washers
1x	RI 17 Stainless steel ring set for sample preparation
1x	TCH Tube clip holder for stage lid
50x	16mm glass cover slips for sample preparation (W16G)
50x	22mm glass cover slips for stage windows (W22G)
10x	22mm x 0.3mm glass cover slips (W22G0.3)

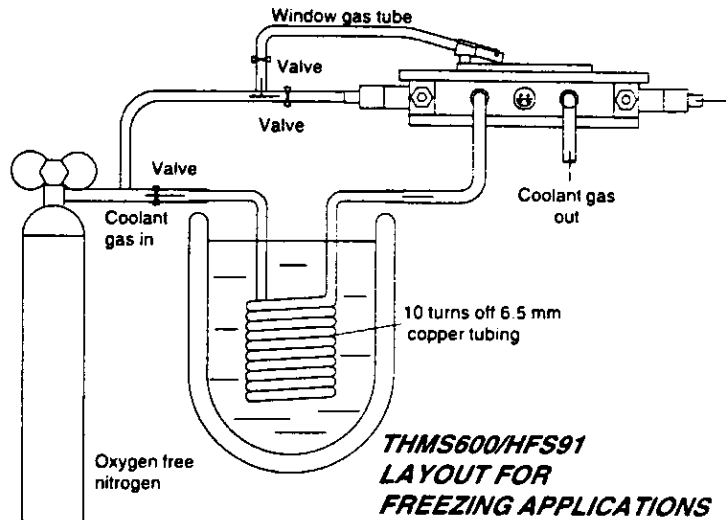
PROGRAMMING AT SUB-AMBIENT TEMPERATURES WITH A COPPER COIL AND DEWAR FLASK

To utilise the full sub-ambient range of the Linkam programmers the coolant gas must be pre-chilled before entering the thermal block. This can be achieved most efficiently by passing oxygen-free nitrogen gas through a series of copper coils immersed in liquid nitrogen.

WARNING: IF COMPRESSED AIR IS USED IT MUST BE COMPLETELY DE-OXYGENATED TO AVOID THE BUILD UP OF POTENTIALLY EXPLOSIVE LIQUID OXYGEN IN THE COOLING COILS

A normal use pre-chilled nitrogen can be used for temperatures down to -180°C .

If liquid nitrogen is pumped into the stage, temperatures as low as -196°C can be achieved in about 20 seconds. The action of the coolant is so effective a much reduced flow of gas compared to that used for cooling at temperatures greater than ambient will be required to obtain the desired cooling rate. If over-cooling occurs the "LOAD" light will remain permanently on, resulting in a loss of control. When heating from sub-ambient temperatures it is possible that the selected heating rate could be too slow for the flow of coolant gas resulting in the 'LOAD' led being permanently off, again resulting in a loss of control. Therefore the flow of coolant gas must be made to balance the effect of the programmer's heating.



PROGRAMMING AT SUB-AMBIENT TEMPERATURES WITH THE LNP COOLING SYSTEM

Description

The LNP cooling system can be used in conjunction with the THMS 600 or HFS 91 stage to cool a sample down to below -94°C .

Wherever possible the small 2 litre desktop dewar flask with the fitted pipe (D2L) should be used, as faster cooling rates can be achieved. For longer experiments or where the supply of liquid nitrogen makes it more convenient to use a larger vessel the DS1 Dewar syphon can be used.

Both devices work by drawing liquid nitrogen from the flask through a very small bore tube via a filter, to prevent blockage from ice particles, where it vaporises nitrogen gas is then passed into a valve which allows manual control of the flow rate. A small quantity of air is also passed through the window gas tube to prevent condensation on the top surface of the lid window.

Operation

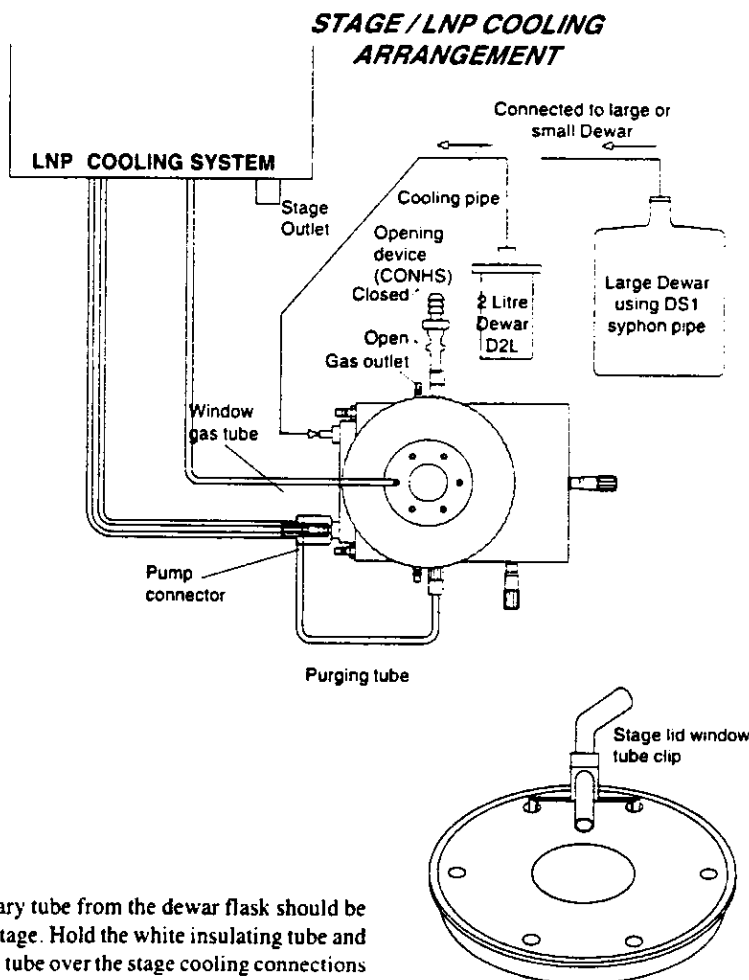
Filling the Dewar

When the dewar is filled, the lid should be placed on before the liquid nitrogen stops filling. When this has finished clamp the lid to place. This is a precaution to prevent the water vapour in the air condensing in the liquid nitrogen.

Connecting the pump

Both the connectors from the dewar flask and the LNP are simply pushed onto the cooling connections of the stage. The black capillary tube from the dewar flask should be inserted inside one of the two cooling tubes on the stage. Hold the white insulating tube and using a pushing and twisting movement position the tube over the stage cooling connections until it comes to a stop (approx 10 mm).

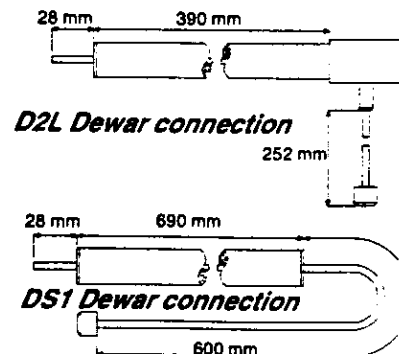
The smaller tube from the outlet marked WINDOW on the LNP should be placed in position on the top of the lid using the clip provided.



Precautions

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. Do not draw air through the stage when it is below room temperature as problems with condensation and icing may occur. Care must be taken not to damage or tightly bend the capillary tube as this will reduce the performance of the unit.

NOTE: UNDER NO CIRCUMSTANCES SHOULD THE TUBE LENGTHS BE CHANGED !



Using the LNP COOLING PUMP

The current speed of the cooling pump is shown on the front panel by a row of red led's on the LNP. The SPEED+ and SPEED- keys are used to select the required setting and therefore the flow of liquid nitrogen through the silver block in the stage.

For minimum liquid nitrogen use and for the best temperature control, the speed should be kept as low as possible. Typical speed-holding are: speed 1 for ambient to -50°C, speed 2 for -50° to -100°C and speed 3 for temperatures below -100 °C. The speed will be changed according to the cooling rate used and the level of liquid nitrogen in the dewar vessel. The extra speeds provided by the LNP enable much faster cooling rates to be achieved - up to 130°C/min as well as lower liquid nitrogen consumption when used in automatic mode. The LNP pump speeds are set as shown below.

STEP		SPEED LED'S
Switch on the LNP. None of the speed LED's should be on.		□ □ □
To increase the speed of the pump press the SPEED+ key. The first speed LED will come on.		■ □ □
Each press of the SPEED+ key will increase the speed of the pump providing more coolant to the stage.		□ ■ □
The maximum speed is shown when the last led is on.		□ □ ■
To reduce the pump speed press the SPEED- key.		□ ■ □
To reduce the pump speed to zero press the SPEED- key a number of times until there are no speed led's on.		□ □ □

LNP OPERATION

Switch on the LNP and the MANUAL led and the speed LED's should be off. With the led off the LNP is in automatic mode with it's speed set by the programmer. The programmer only sets the speed when the temperature is less than 100°C. It may also switch the LNP off when heating from sub-ambient temperatures at high heating rates, as it is no longer necessary to have a flow of coolant in the stage. To switch back to manual mode press the MANUAL key, the led will come on and if the pump is already on it will be switched off to allow the user to manually set their own speed.

NOTE: When used with the LinkSys Windows software the MANUAL switch becomes in-operative.

COOLING PROCEDURE FOR STAGE AND LNP COOLING PUMP

If the stage is to be used at low temperatures the stage clamps should always be used if possible. This prevents the stage from moving when the cooling pipe from the Dewar becomes less flexible due to the liquid nitrogen flow.

- THMS 600 ONLY:** Open the end door of the stage and insert the sample holder. Ensure that the sample carrier slides smoothly down the ramp and onto the silver block. The ring should lie as flat as possible on the block so that the quartz disc will locate correctly. Adjust the manipulators so that the block window is in the centre of the ring.
- THMS 600 with G7-10 ONLY:** Use the vacuum tweezers to place a 7 or 10 mm quartz disc under the ring of the sample carrier.
HFS 91 with G7-10 ONLY: Use the vacuum tweezers to place a 7 or 10 mm quartz disc onto the middle of the silver block.
- Use the vacuum tweezers to place a sample onto the quartz disc. Check that the sample does not touch the sample carrier ring as this can cause temperature errors.
- Silver Cover ONLY:** Place the silver block cover over the sample. For a THMS 600 position the sample holder (G7 or G10) in the centre of the silver block, the silver block cover fits over the block with the neck of the sample holder situated squarely in the side slot of the silver block cover to allow movement of the sample carrier.
- Place a 13 mm glass cover slip over the top of the silver block cover which prevents condensation forming on the sample.
- Screw the stage lid onto the body.
- Connect the cooling system as shown on the previous page. Connect the purging tube to the stage using the quick release gas connector provided. (SEE STAGE / LNP COOLING ARRANGEMENT DRAWING ON PREVIOUS PAGE).

The gas outlet on the inner side of the stage should be opened by inserting the valve insert, CONHS. Place the window gas tube into position on the top of the lid using the clip provided. The clip should be positioned in one of the six holes such that the lens turret of the microscope can be swung freely around. The end of the tube must be facing the centre of the stage lid window. Make sure that the lid is screwed down correctly onto the 'O' ring seals.

- 9) Switch on the programmer and set the limit to 30°C. Press the START button and wait until the limit is reached.
- 10) Switch on the LNP cooling system and when operating in the manual mode set to the maximum speed.
- 11) Block the hole in the pump connector which is on the opposite side to the purging tube and block the window tube, by pinching or folding it, for about 60 seconds. During the 60 seconds, occasionally block the end of the valve insert quickly with your finger. This allows maximum mixing of the Nitrogen with residual air in the stage chamber and hence evacuation of most of the air from the chamber. With the silver block at 30°C any remaining water vapour will condense on the colder tubes which carry the coolant to the block.
- 12) Remove the valve insert (CONHS) and unblock the hole in the pump connector and the window tube.
- 13) After this initial set up, the programmer can be set with the required limit and rate and used in the normal way. Adjust the LNP speed settings until the load light on the programmer is flashing on and off evenly (LNP 93/1 only). When using the automatic LNP 93/2 set the LED on the front of the LNP to automatic and the pump will be controlled by the temperature programmer.

NOTE: If the stage is dismantled for any reason, special care should be taken to ensure that the 'O' ring seals are replaced correctly. (see THMS600/HFS91 BOTTOM WINDOW ASSEMBLY)

LNP 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 LNP. 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.*

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.*

Re-align the window gas tube clip to the required position in the stage lid.

USING THE STAGE WITH 80 AND 100X LENSES

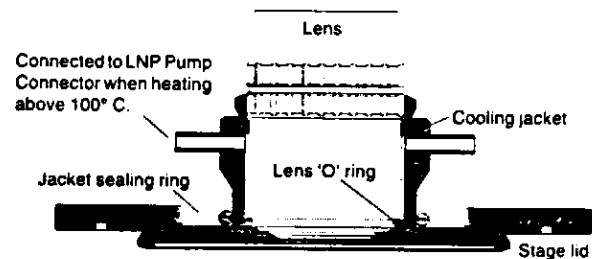
When using the stage with super long working distance lenses a specially designed lid and cooling jacket is required. The lens is placed inside the cooling jacket which is then inserted into the lid of the stage. To increase the optical performance of the stage, the lid is sealed to the cooling jacket using 'O' rings, and therefore the lid does not have a window in it. Once the chamber is purged with nitrogen gas there will be no condensation.

100 X Lens

It is recommended that the Nikon F2 trinocular head is used as this ensures that there is minimum glass between the sample and the objective. The microscope analyser should also be removed to ensure optimum resolution of the sample.

To assemble the jacket and lid follow the steps below:

- 1) Mount the stage on the microscope's sub-stage using the appropriate clamps. Using a 10X lens focused on the window of the silver block, adjust the stage position until the window is centred.
- 2) Adjust the condenser lens as described earlier.
- 3) Place the sample on the silver block.
- 4) Remove the lid from the stage body and check that the jacket sealing 'O' ring is in place.
- 5) Unscrew the two halves of the cooling jacket and make sure that the lens 'O' ring seal is in place. Unscrew the lens from the microscope and place it inside the jacket. Place the other half of the cooling jacket over the lens and screw the jacket together.
- 6) Tighten the jacket sealing ring until it just starts to pinch the 'O' ring. Push the cooling jacket assembly into the lid. It should be possible to pull out and push in to the lid smoothly and without too much force. If the jacket sealing ring is too tight the 'O' ring will bow and the seal between the jacket and lid will be lost.
- 7) Pull the assembly out of the lid and screw the lens into the microscope.
- 8) Screw the lid onto the stage. Gently move the stage up, until the cooling jacket is fully inside the jacket sealing ring. Adjust the focus.
- 9) Follow the cooling procedure outlined earlier.

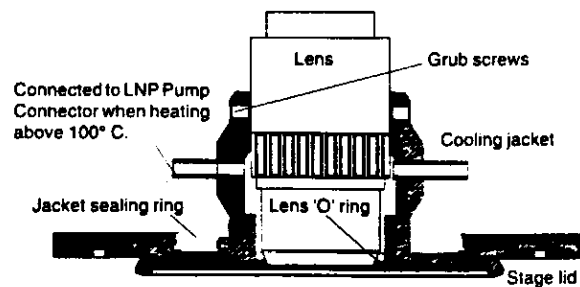


NOTE: Each time the cooling jacket is removed from the jacket sealing ring, or if a new sample is loaded the system will require re-purging.

80 X Lens

To assemble the jacket and lid follow the steps below:

- 1) Follow steps 1 to 4 from the 100x lens setup.
- 2) Unscrew the lens from the microscope and place it inside the jacket until it comes to a stop. Gently tighten the grub screws to hold the lens in place.
- 3) Follow steps 6 to 9 from the 100x lens setup.



PRECAUTIONS FOR USE WHEN HEATING ABOVE +100°C

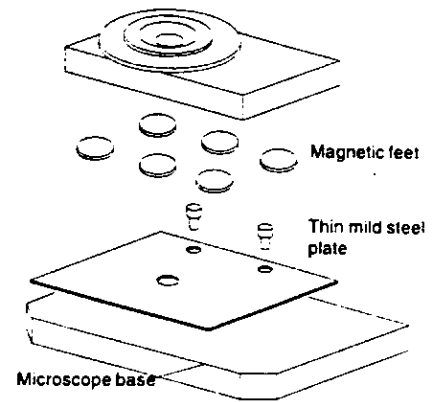
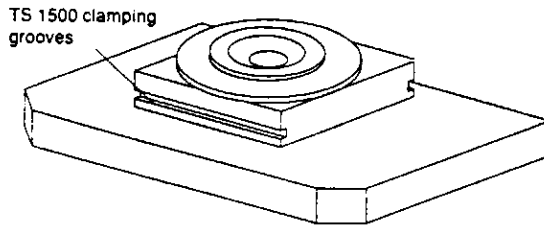
As the stage lid does not contain a window, the lens must be cooled using the cooling jacket and pump when heating the THMS or H 91 Stage above 100°C. This is achieved by removing the pump connector from the Stage cooling pipes and connecting it instead to the pipe on the side of the cooling jacket.

THIS MUST BE DONE IN ORDER TO AVOID ANY DAMAGE TO THE LENS.

TS 1500 STAGE

Microscope Mounting

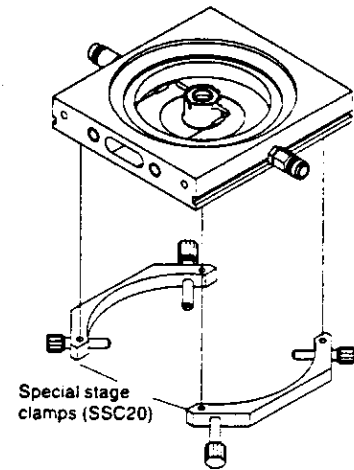
Six self adhesive magnetic feet are supplied which can be used together with a thin mild steel plate to attach the hot stage to the microscope's mechanical base. The TS 1500 can also be mounted using the clamping grooves on the side of the body. If it is not practical to use the magnetic feet, double-sided adhesive tape can be used.



Mounting Arrangement for Nikon and Olympus Microscopes

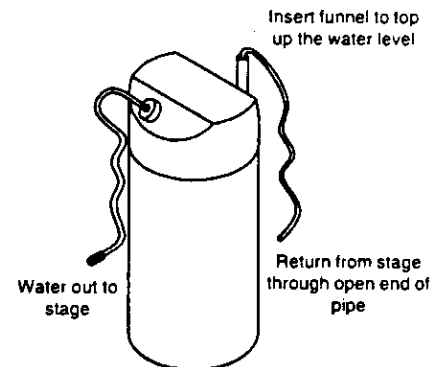
This arrangement uses a set of SSC20 stage clamps which are simply screwed to the bottom of the existing TS 1500 body using four cap screws which are provided. The four positioning screws should be used to centre the hole in the ceramic cup with the microscope's light source as follows.

- 1) Loosen the four screws and position the stage and clamps on the microscope's sub stage.
- 2) Select a 10X lens and focus on the edges of the ceramic cup's hole.
- 3) Gradually adjust the screws until the hole is central to the light path.



Stage Water Cooling Circulator Assembly

Assemble the water circulator by pushing the green flexible tubing onto the tapered pipe at the bottom of the reservoir. Secure the tubing in place by screwing the collar over the tube and onto the fitting.



Circulator Filling

The water circulator should be filled at a sink as follows :

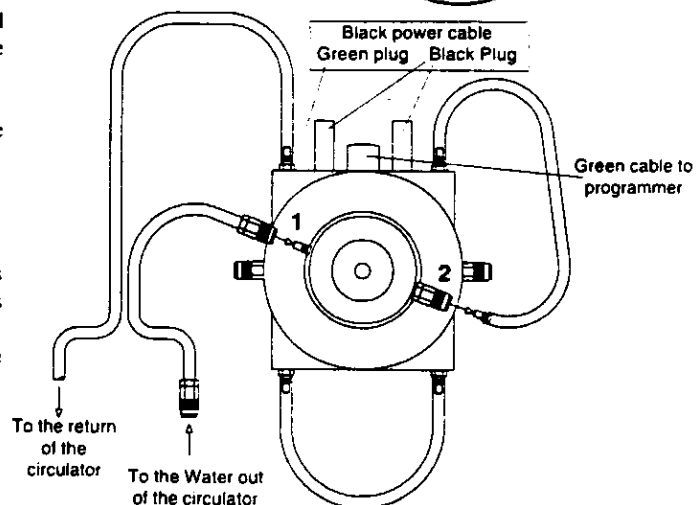
- 1) Remove the pump unit from the top of the water chamber.
- 2) Fill the water chamber until the level is 50 mm below the rim.
- 3) Replace the pump unit carefully as the 'O' ring is a tight fit and water may spurt out of the stage tube.
- 4) Top up the water level using the funnel provided until the water level in the tube is higher than the top of the pump.
- 5) Connect the pump to the stage as shown.
- 6) Place the water circulator on the floor to ensure that the water level is below the stage.

Circulator Connection

The water cooling for the TS 1500 **must** be connected as shown here to prevent water from escaping when the lid tubes are removed during sample loading.

To remove the lid while the pump is still running - remove connector 1 and then connector 2.

Reverse this sequence once the lid has been replaced.



Electrical Connections

The thermocouple sensor inside the TS 1500 is connected to the programmer through a green connector and cable which is permanent attached to the rear of the programmer. The thermocouple connector on the stage has two rectangular pins, one of which is wider than the other. Make sure that the pins on the stage are mated correctly with the socket on the cable.

The black power cable with the 'D' type connector and the three 4 mm plugs connects the power from the programmer to the stage. The black plugs connect to the two sockets mounted in white PTFE and the green plug to the socket mounted directly in the body. These are sized differently in order to avoid a connection problem.

Using the TS 1500 stage

To prolong the life of the stage, try to avoid consistently high heating rates and temperatures as this can lead to excessive grain growth in the platinum heater with the resulting effect that the wire becomes thinner and finally fails.

Above 300°C, if the EXIT key is pressed or if the end of profile is reached, the TMS 93/1500 will cool at 200°C/minute down to 30 before shutting off. This prevents the sudden drop in temperature which could cause the ceramic cup and its supports to crack. programmed cool to 300°C should always be used.

Sample Preparation

The TS1500 responds very quickly to changes in temperature on the sample, so to prevent any damage to the heater it is strongly advised that no COLD samples be introduced at higher temperatures and that the sample be as thin and as light as possible (<270 mg).

Samples must always be placed on the W7S sapphire disc or a quartz disc as the sample can fuse into the ceramic cup or can attack the thermocouple causing incorrect temperatures and leading to a failure of the heater.

For the same reasons quartz discs should not be used at temperatures >1000°C.

If the samples give off volatiles they should be removed by sucking them out through one of the gas connectors.

Gas flow into the stage should be limited to about 60 cc/min as flows greater than this can cause problems with the temperature control and the accuracy of the temperature measured. Thermally conductive gases such as helium can also effect the temperature control and limit temperature accuracy.

If the quartz window in the lid becomes dirty or stained it may be cleaned and polished using a small quantity of jewellers rouge.

Stage heater protection

The TS 1500 stage is protected in the programmer by a solid state cut-out. If for any reason the current into the heater exceeds a pre-level the power will be automatically turned off and the message 'PWR SURGE' will be displayed on the programmer. This could happen for instance if a cold sample is introduced at high temperatures or if a gas flow was too high.

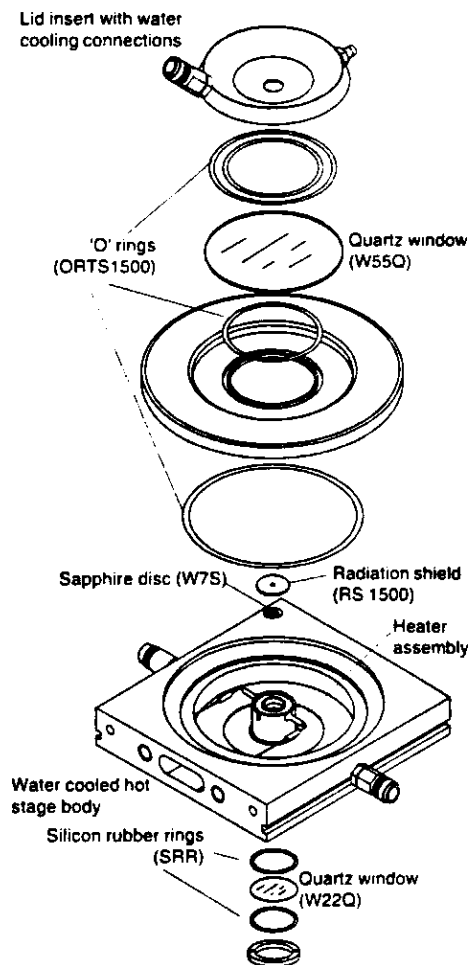
To reset the programmer allow the stage to cool and then press any key on the programmer.

TS 1500 HEATER AND LID ASSEMBLY




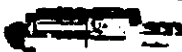
The diagram shows how the quartz windows are fitted into the TS 1500 and if necessary how the element can be removed for repair.

TS 1500 SPARES




SPARE ELEMENT FOR TS1500 STAGE	SET1500
RADIATION SHIELD	RS1500
SET OF O RING SEALS FOR STAGE BODY AND LID	ORTS1500
55 mm BY 1 mm QUARTZ WINDOW FOR TS1500 TOP LID	W55Q
22 mm BY 0.5 mm QUARTZ WINDOW FOR TS1500	
BOTTOM WINDOW	W22Q
7 mm BY 0.3 mm SAPPHIRE SAMPLE DISC FOR TS1500	W7S






CONNECTORS FOR WATER AND GAS

Code	Description	
CONHS	Water hose connector	
CONTS	Threaded straight connector	
CONTV	Threaded valve connector	
CONHV	Hose valve connector	

CRUCIBLES AND CARRIERS

FTIR/D	2.5 mm ^ø by 0.25 mm Diamond window mounted in 7mm ^ø silver disc	
G7T G7	7 mm diameter tapered sample carrier 7 mm ^ø FT-IR sample carrier	
G10T G10	10 mm ^ø tapered sample carrier 10mm FT-IR sample carrier	
LTS/CC	LTS 350 crucible carrier	
THMS/CC THMSG/CC	Crucible carrier for THMS 600 Crucible carrier for use with THMSG 600	
THMS/LCC THMS/Q THMSQ/VOL THMSQ/VOL/C THMS/S	Liquid crystal cell carrier for THMS 600 Quartz crucible for THMS/CC 18 mm ^ø Polished quartz crucible with cover (15 mm ^ø I.D. X 0.5 mm) for THMS/CC 18 mm ^ø Cover for polished quartz crucible (THMSQ/VOL) Stainless steel crucible with a floating 15 mm sapphire window for THMSG/CC	



LNP COOLING PUMP AND DEWAR SPARES

DF1	1litre Dewar and copper coil for fast cooling	
DF2	2litre Dewar and syphon assembly	
D2L	Dewar syphon assembly (no dewar lid)	
DS1	Dewar syphon for use with large dewar vessels	
DPC	Connector for PVC tubing between block and LNP	
DF2GI	2 litre Dewar flask insert	
DF2TU	White insulation tube for Dewar (per metre)	
SIL6	Silicon tubing 6 mm O.D. 3mm I.D (per metre)	
TCH	Tube clip holder for stage	

MISCELLANEOUS

CSCO2	Temperature Calibration standard for -56.6°C
CSKNO3	Temperature Calibration standard for 333°C

MICROSCOPE MOUNTINGS

MGF	Set of 6 magnetic feet for stage mounting	
SSA 20	Stage adaptor for Zeiss Axiophot and Axioplan	
SSA 30	Stage adaptor for Zeiss Axioskop & Axiolab	

SSA 40

Stage adaptor for Leica Microscope

SSC 10

Stage clamps for Nikon and Olympus Microscopes



SSC 20

Clamps for attaching the TS 1500 to Nikon & Olympus Microscopes



STAGE SPARES AND ACCESSORIES

- LE2 Stage Connector for external electrical signals (liquid crystal)
- ORTHMS Set of 'O' ring seals for the THMS stage body and lid
- ORTS1500 Set of 'O' ring seals the TS 1500 stage body and lid
- PT100 Platinum resistor 1/10 din 1.6 mm X 15 mm
- RI17 Stainless steel rings for sample arrangement

SCO

Silver block lid



SRR

Silicon rubber rings for top and bottom stage windows

STLID

Stage Lid (state which type of stage)

Replacement Heaters

- SET1500 Heating element for TS 1500
- SET1500ex TS 1500 heating element exchange for old element

THMSB

Replacement heater assembly for THMS 600

THMSGB

Replacement Geology heater assembly for THMS 600



VACUUM TWEEZER SPARES AND ACCESSORIES

AP8 Vacuum pump and tweezers with large rubber suction cup

VT10

Vacuum tweezers for mouth operation with small rubber suction cup

VTM

Mouthpiece for VT10

VTRL

Large rubber suction cup for VT10

VTRS

Small rubber suction cup for VT10

SLS

Sample loading station

WATER CIRCULATOR

ECP220 Water Circulator for use with 220-240V a.c. mains

ECP110 Water Circulator for use with 110-130V a.c. mains



WINDOWS

LCC5 Liquid crystal cell - 5um AP Capillary fill, 4 electrode

Barium Fluoride

W7BaF2 7mm x 0.5 mm Barium Fluoride for G7 sample holder
W10B 10mm x 0.5 mm Barium Fluoride for G10 sample holder
W22B 22mm x 0.5 mm Barium Fluoride for G22 sample holder

Glass

W13G 13mm x 0.1mm glass cover slips for silver lid assembly (box of 100)
W16G 16mm x 0.17 mm glass cover slips (box of 100)
W22G 22mm x 0.17mm glass cover slips for top window (box of 100)
W22G0.3 22mm x 0.3mm glass cover slips for bottom window (box of 100)

Potassium Bromide

W7K 7mm x 0.5 mm Potassium Bromide for G7 sample holder
W10K 10mm x 0.5 mm Potassium Bromide for G10 sample holder
W16K 16mm x 0.5 mm Potassium Bromide for G16 sample holder
W22K 22mm x 0.5 mm Potassium Bromide for stage lid and bottom window

Quartz

W600Q 2.4mm x 1.6 mm quartz window for the silver block heater assembly
W7Q 7mm x 0.3mm quartz window for G7 sample holder
W7TQ 7mm x 0.3mm tapered quartz window for use with G7T sample holder
W10Q 10mm x 0.3mm quartz window for use with G10 sample holder
W10TQ 10mm x 0.3mm tapered quartz window for use with G10T sample holder
W16Q 16mm x 0.3 mm quartz window for G16 sample holder
THMSQ/VOL/C 18 mm^ø Cover for polished quartz crucible (THMSQ/VOL)
W55Q1 50 mm^ø X 1 mm quartz window for TS 1500 lid
W55Q1.8 50 mm^ø X 1.8 mm quartz window for TS 1500 lid (high vacuum)

Sapphire

W7S 7mm x 0.3mm sapphire window for G7 sample holder and TS 1500 sample placement
W7TS 7mm x 0.3mm tapered sapphire window for use with G7T sample holder

Zinc Selenide

W7Z 7mm Zinc Selenide window
W22Z 22mm Zinc Selenide window

OPTICAL ACCESSORIES

MD300 Microscope graticule for Lens calibration (10*10um and 10*100um)

SLO 80 Special lid and cooling jacket for Olympus x80 ULWD lens
SLO 100 Special lid and cooling jacket for Olympus x100 μ lens
SLN 100 Special lid and cooling jacket for Nikon x100 SLWD lens

