CAP 500
Capillary Pressure Stage

USER GUIDE

Introduction

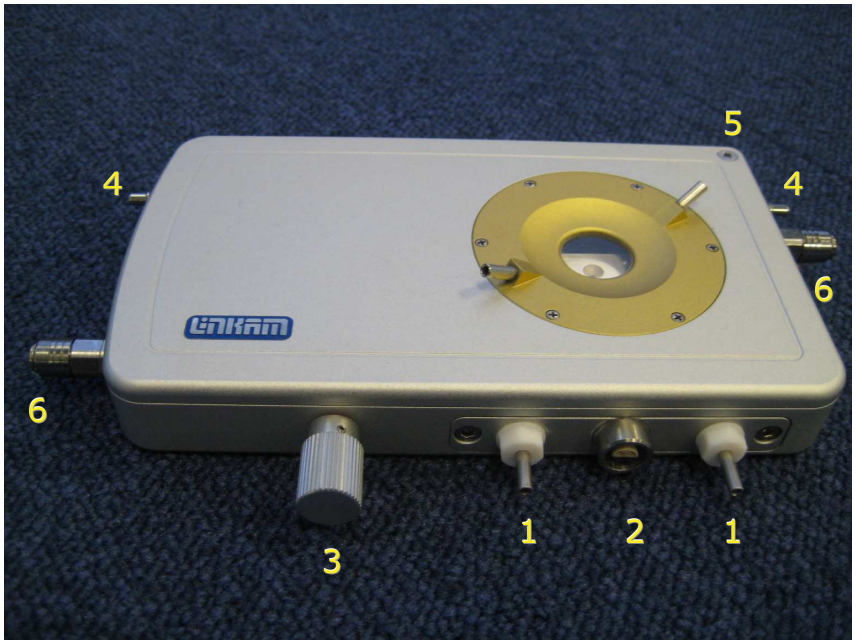
The CAP500 stage is designed to heat or cool 1 by 1mm wide, 0.6mm deep channel inside a 50mm silver block. Capillaries loaded into this channel can be moved 12.5mm from the centre (a total of 25mm movement).

The stage is sealed with an O'ring so that purging gas can be used to prevent condensation during cooling.

When heating samples above 300°C for longer than 30 minutes, the stage body can be water cooled.
The stage lid has a simple hinged mechanism in one corner.
The lid can be removed by lifting the lid when it is open.

The silver block can rapidly heat and cool in the range of –196°C to 500°C at a rate from 0.1 to 50°C/min
The block has been designed to minimise the temperature gradient along the length. A test setup with results are shown later in this manual.

The temperature sensor mounted in the silver block is a high accuracy 100ohm platinum resistor to 1/10th Din Class A and is accurate to 0.1°C.



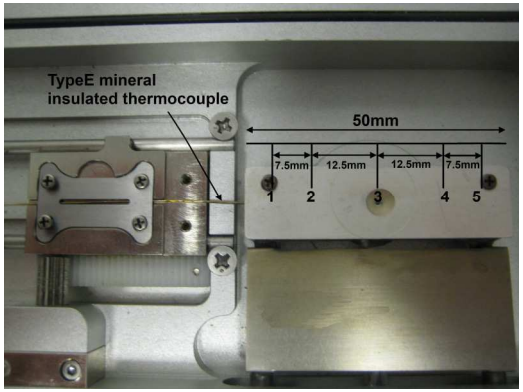
Control stability using the T95 System Controller is better than 0.1°C over the entire range.

1) Liquid nitrogen inlet/outlet
2) T95 System Controller connection
3) Capillary movement control (25mm)
4) Stage body water cooling
5) Lid hinge
6) Gas in/out (purging)

Results

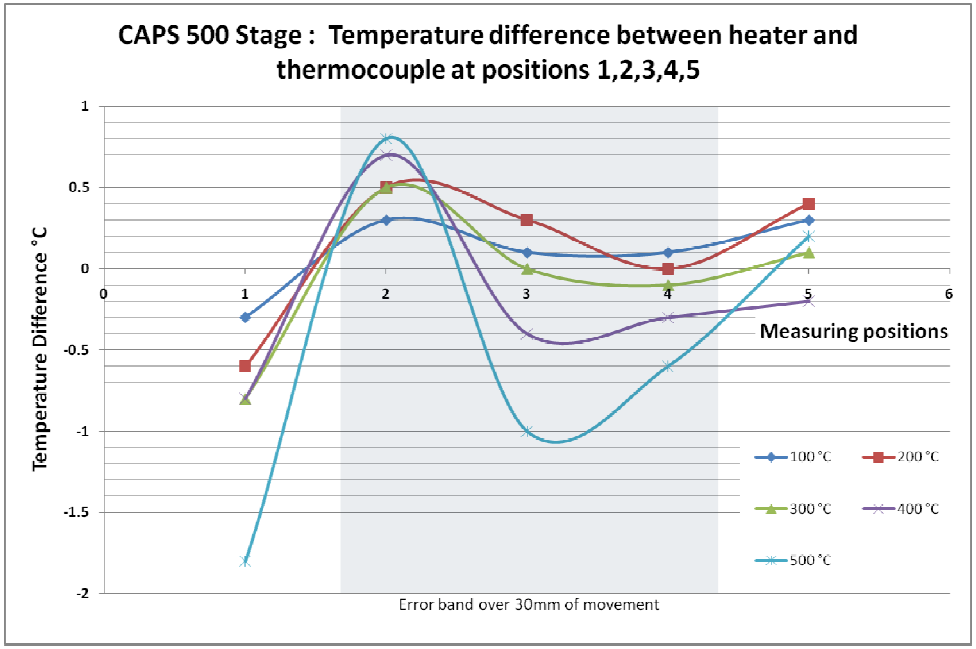
The results with this thermocouple should only be treated as a trend indicator rather than accurate temperature values.

The thermocouple was mounted using the same sample holder as the capillary in order to achieve the best simulation. Measurements were made at points 1, 2, 3, 4 and 5 (see picture) at temperatures of 100°C, 200°C, 300°C, 400°C, and 500°C. These results are shown below.
NOTE: The highlighted band on the graph represents the 25mm of sample movement. i.e the sample will not experience the gradient at points 1 and 5



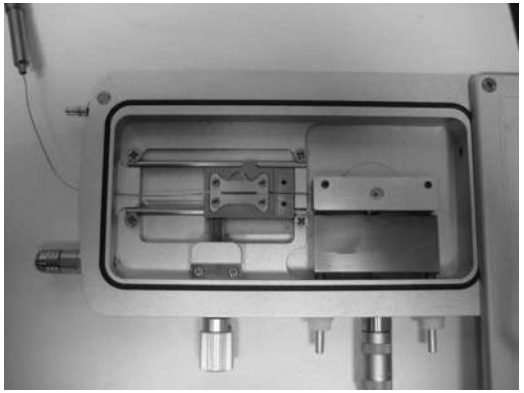
CAP500 Stage temperature difference along heater

Stage °C	Temperature in °C at marked points				
	1	2	3	4	5
100	99.7	100.3	100.1	100.1	100.3
200	199.4	200.5	200.3	200	200.4
300	299.2	300.5	300	299.9	300.1
400	399.2	400.7	399.6	399.7	399.8
500	498.2	500.8	499	499.4	500.2



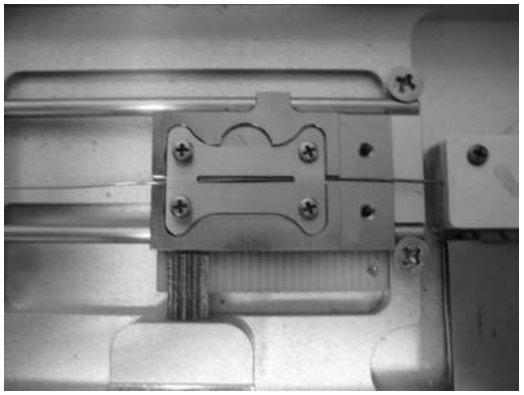
Setup to determine the temperature gradient

To test the gradient of the block over the length of the heater, a small 0.5mm diameter mineral insulated Type E thermocouple was used with an ungrounded junction and mounted in the same sample holder as the capillary in order to achieve the best simulation.



The thermocouple is available from Omega Engineering Ltd (www.omega.com) part number HEMTSS-020(U)-6 and is manufactured with special limits of error wire inside a stainless steel sheath.
Although the materials are manufactured to a close tolerance it can yield errors of 1.0°C or 0.4% (whichever is the greater) above 0°C. At 500°C this could result in an error of 2.0°C.

TypeE is a Nickel-Chromium vs Copper-Nickel thermocouple and has the highest output voltage (68uV/°C at 100°C) of all the noble and base metal thermocouple types.



Thermocouple voltage is actually generated by the section of wire that contains the temperature gradient and not necessarily by the junction itself. **In this experiment we have a region of the thermocouple probe inside the isothermal block, but a another region is outside the block and has a large gradient. In addition the sheath of the thermocouple will also act as a heat sink.**

Importance of embedding the thermocouple

An example of this gradient effect of the thermocouple is to measure again at point 1, ***but with the thermocouple entering the block at the other side so a longer section is at an isothermal temperature.***
This yields a value of 500.3°C instead of the 498.2°C recorded in table on page 7.

Temperature calibration results for the thermocouple were carried out at liquid Nitrogen and with boiling water.

Liquid Nitrogen (-196°C) showed -193°C.
Boiling water (100°C) showed 100°C.

Silver Block Temperature Accuracy

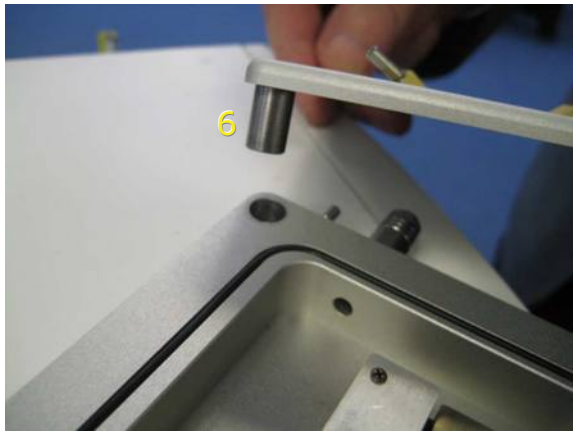
A capillary was filled with distilled water and cooled to –20°C. On heating at 2°C/min the melting point onset was seen at 0.0°C.

Inside the CAP500

7) Capillary sample holder
8) Capillary movement mechanism
9) Silver block heater and cover
10) Platinum sensor protection plate
11) 16mm glass cover slip over the heater aperture

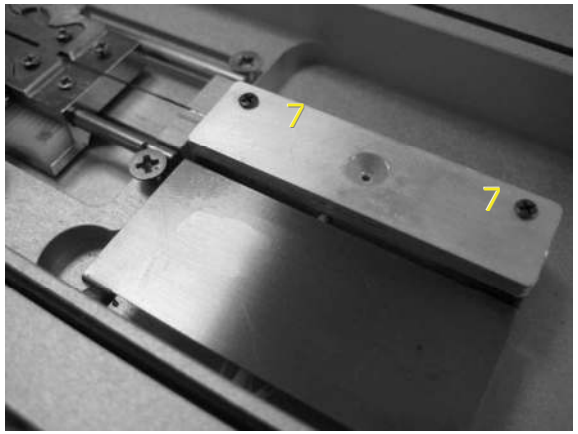
Lid Removal

The lid can be removed if necessary, by rotating the lid to the open position and simply lifted it away from the body.
To refit, push the hinge pin (6) back into the body.
The magnets will hold the lid in the open or closed positions.



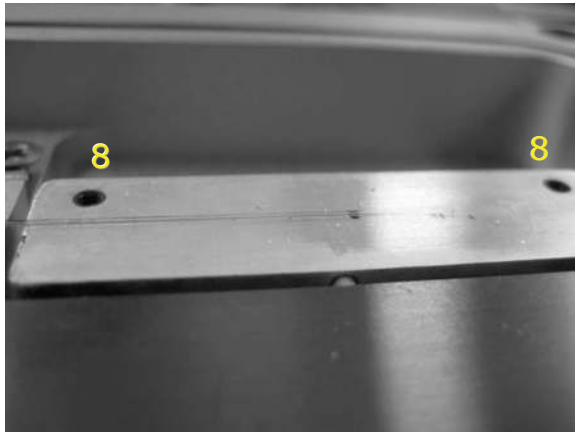
Silver Heater Block Cover

The heater cover can be removed using the two screws (7).
Do not over tighten these when refitting the lid.
Always use a 16mm glass cover slip over the heater aperture to improve the temperature accuracy.



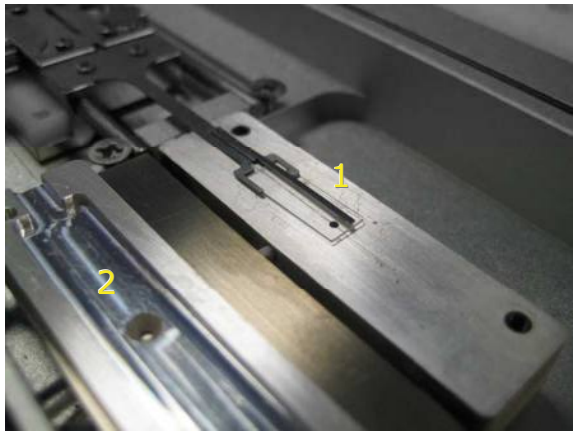
Capillary Alignment

If the heater block needs to be re-aligned with a capillary of a different size, remove the block cover and adjust the two allen screws until the capillary tube is flat as shown in the picture.
Note: Clockwise will lift the block, anti-clockwise will lower the block.



Quartz Sample Carrier

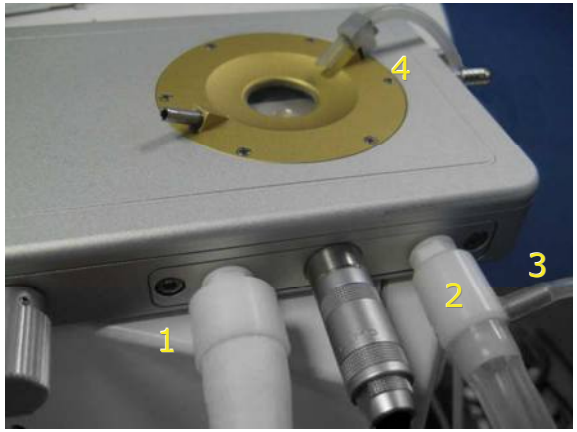
Instead of using the capillary, a quartz sample carrier (1) can be mounted to the sample holder to give 25mm of movement.
This requires a different silver cover (2) to be used.
Do not over tighten these when refitting the lid.



Liquid Nitrogen 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 one of 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).

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 (4). This tube blows warm recycled nitrogen gas across the lid window to prevent condensation on the viewing window surface.