General Guide to the Linkam THMS600 Stages APS Detector/Equipment Pool

Table of Contents

- Quick Guide
- Safety and Handling
- THMS600 Connections and Parts
- LN2 Sample Cooling
- Water Cooling for Stage Body
- Software Controls



Reference: much of the information in this guide is gleaned from User Manuals provided by Linkam with purchase of equipment. If more information is needed, please contact Detector Pool staff at 2-9493 or dp@aps.anl.gov.

The Linkam THMS600 stage can be used to heat or cool a sample from -196°C to 600°C at speeds of 1-100°C per minute. The Detector Pool has one older stage as well as a newer stage with an updated controller design.

Quick Guide - 1

Safety & Handling

- Linkam stages have **EXTREMELY** fragile platinum leads; please do not touch or move.
- When heating above 300°C, stages require water cooling. Contact DP staff (2-9490).
- Regarding gas purging:
 - Do not use hydrogen or helium (thermal conductivity)
 - Dilute any reactive gases with 95% inert gas
 - Use only mixtures containing less than 20% oxygen
 - Flow less than 60 CC/min
- If using LN2 sample cooling, please follow all APS procedures for safe handling of LN2.



- The black capillary tube on the LN2 dewar lid is fragile; please handle carefully.
- Disconnect the LN2 dewar from the stage before heating above 300°C.

Hardware Operation

Make all connections





- If cooling sample with LN2:
 - Fill the dewar approximately 2/3 full.
 - The LNP95 must be switched on before the T95/CI95 system controller.
 - The stage chamber needs to be purged of air before starting a cooling experiment.

Please review the complete User Guide to ensure safe and effective operation.

Quick Guide - 2

Software Operation

- Turn on the computer, and log in
 - Username = dpuser
 - Contact DP staff for the password
 - Or, use the sector's LDAP account
- The start-up screen (lower left) will appear
 - Select "Linkam from the dropdown menu
 - Click "Start" to start the IOC and medm
- Use the EPICS control screen (lower right) to set rate (°C/min) and desired temperature.
 - Pump speed is generally set to "Automatic."
 - To adjust the y-axis on the strip chart display, rightclick, then select "PV Limits."

🔲 DP EPICS Launcher - Master 💷 🗆 🗙		×
Help Documents Detector Pool	LINKAIII CI94	
Select Detector	Cooling Rate too fast Scan Bisable Open Circuit Power Surge Puep Control No FV1 200	
Linkam	Both Stages Automatic at	
Mar345	Set Value Current Value Start	
Prosilica	Rate 0.00 Hold	
Vortex	Temperature 1 0.0 28.00 Clear Buffer	
Linkam	DSC value 0 End After Profile	
	Status Stopped End On Stop	
DP Linkam Startup _ X	8 6.0 (emp (c)	
Linkam	1 0.0 1 4.0 1 6.0	
IOC Start Stop	Print Close PV Info Use PV Lints PV Lints	
MEDM Start Stop	List Topologic Hidden Button Harkers	

Sample Loading



















Aluminium and sapphire crucibles

Tzero Press, often used with DSC

Safety and Handling

- The heating element may be fragile. Exercise care when loading and unloading samples.
- Use as little sample as possible to reduce thermal load and avoid damaging the heating element.
- Heating over 300°C
 - Stages require water cooling when heated above 300°C. Contact DP staff (2-9490 or <u>dp@aps.anl.gov</u>) to obtain water circulator.
 - Disconnect the LN2 dewar from the stage before heating above 300°C. Leaving the tubing connected can damage the tubing and/or heater.
- Regarding gas purging:
 - Do not use hydrogen or helium (due to high thermal conductivity)
 - Do not use a gas mixture containing more than 20% oxygen
 - Dilute any reactive gases with 95% inert gas such as nitrogen or argon
 - Use a gas flow of less than 60 CC/min to avoid overloading the heating element
- Failure to observe these guidelines may result in heating element failure.
 - If you plan to use the stage in a manner not recommended above, please purchase your own heating element.
- If using LN2 sample cooling, please follow all APS procedures for safe handling of LN2.

Connections for THMS600 (old) with & w/o cooling



Crossover/null modem cable connects to computer serial port. I²C cable connects CI94 to LNP LN2 dewar connects to stage; securely connect inner tube first.

Connections for THMS600 (new) with & w/o cooling



Crossover/null modem cable connects to computer serial port.



I²C cable connects CI94 to LNP LN2 dewar connects to stage; securely connect inner tube first.

THMS600 Parts

Lid Window Assembly

To replace the windows in the Stage Lid (1) use the Window Tool (2) and align the two wide spacing pins to the Tube Clip Holder holes and unscrew the Lid Insert (3). The Stage Lid and Lid Insert should be turned upside down as shown in the diagram opposite and reassembled in the order indicated.

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



Bottom Window Assembly

Use two narrow spacing pins of Window Tool (2) to align it to the two holes of Window Locking Ring (4) and unscrew. Reassemble the bottom window as shown in the opposite diagram



15. Heating element wire
16. Stainless steel cooling tube
17.22mm diameter pure silver heating block
18. Platinum temperature sensor
19. Sample holder ramp
20. Aperture hole

21. Vertical Sample Holder

Stage Assembly

- 1. Lemo connector for stage lead
- 2. Heating element carrier assembly
- 3. Stage body
- 4. Stage body water connector
- 5. Gas purge valve
- 6. Y-Sample manipulator
- 7. Stage door
- 8. Door locking thumbscrew
- 9. X-Sample manipulator
- 10. Liquid nitrogen cooling connector
- 11. Bypass stage body water cooling connector
- 12. Sample chamber

13 Farth safety contact for lid



Photos/graphics courtesy of linkam.co.uk

Sample Cooling with LNP95

- If using LN2 cooling, please follow all APS procedures for safe handling of LN2.
- The black capillary tube on the dewar lid is fragile. Please handle carefully.



- Fill the dewar approximately 2/3 full.
- The LNP95 must be switched on before the T95/CI95 system controller.
- The stage chamber needs to be purged of air before starting a cooling experiment.
 - See purging procedure on next page
- Disconnect the LN2 dewar from the stage before heating above 300°C. Leaving the tubing connected can damage the tube and/or heater.

Purging Procedure

- For full details, please see the THMS600 User Guide from Linkam, available on the Detector Pool website.
- To purge using recycled nitrogen gas produced from the Dewar:
 - Make all connections, make sure that the stage lid and gas inserts are secure, turn on the LNP95, then turn on the T95/CI95.
 - Set the LNP95 to "Manual" mode.
 - Set the T95/CI95 to hold at 40°C.
 - Set the LNP95 to a speed of 100.
 - Block the hole in the white plastic pump connector, and pinch the narrow window tube to block it.
 - Block the gas outlet for a few seconds to allow pressure to build, then release.
 Repeat for a few minutes.
 - When finished, unblock the pump connector, window, tube, and gas outlets.
 - Change the LNP95 to "Automatic" mode.



Purging

tube

Pinch window tube with left hand

Connector with finger pump connector

Block and release outlet valve with finger

Photo courtesy of linkam.co.uk

Water Cooling Connections





- When heating above 300°C, stages require water cooling to keep the stage body and lid cool. Contact DP staff (2-9490; <u>dp@aps.anl.gov</u>) for a water circulator.
- There are several options for cooling:
 - Linkam ECP Water Circulating Pump
 - It may be necessary to prime the ECP if the water connectors have been removed.
 - DP circulating pump with custom housing
 - To ensure proper circulation, be careful to avoid introducing air into the system. While connecting and disconnecting, take care to prevent water from leaking out.
 - Beamlines may use their own small, recirculating chiller. (less than 20CC/min flow rate)



EPICS Software Controls

- Turn on the computer, and log in.
 - Username = dpuser
 - Contact DP staff for the password
 - Or, use the sector's LDAP account (see beamline personnel)
- The start-up screen (shown right) will appear.
 - Select "Linkam from the dropdown menu
 - Click "Start" to start the IOC and medm
- The figure below shows the two resulting terminal windows and the Linkam control screen.

dpuser@xraydetector45:/local/DPbin/Scripts _ 🗆 🗙	ci94_new.adl _ 🗆 🗙
File Edit View Search Terminal Help [dpussergaraydetector45 ~]5 cd /local/DPbin/Scripts/	Linkam Cl94
<pre>expert: Command not found.usip: //stml_Loc_Linkam expert: Command not found.usip: //stml_Loc_Linkam expert: Command not found.usip: //stml_Loc_Linkam expectise.stmlp: Command not found.usip: //stml_Loc_Linkam/EPLinkam!') epicisEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam/EPLinkam!') epicsEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam/EPLinkam!') epicsEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam/EPLinkam!') epicsEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam_SI_2.2') epicsEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam_SI_2.2') epicsEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam_SI_2.2') epicsEnset("SUPPORT", 'local/DPDin/epics/epics_2013-02-26/Junkam_SI_2.2') epicsEnset("SUPPORT", 'local/DPDin/epics/epics/epics_2013-02-26/Junkam_SI_2.2') epicsEnset("SUPPORT", 'local/DPDin/epics/epics/epics_2013-02-26/Junkam_SI_2.2') </pre>	Error Status Cooling Rate too fast Open Circuit Power Surge No Exit 300 Both Stages Link Error NC Default
pert/ansabetectorR1.8 ⁻¹), focus of bring proving pro	Set Value Current Value Pump Speed 0 0 Rate 0.00 Hold
1.4°) epicieEnSet("CALC","/local/DPbin/epics/epics_2013-02-26/synApps_5_6/support/calc -3.1°) epicieEnSet("CAMAC","/local/DPbin/epics/epics_2013-02-26/synApps_5_6/support/cam ac-2-7°)	Temperature 1 0,0 28,00 Stop Temperature 2 27,00 Clear Buffer End After Profile
dpuser@xraydetector45:/local/DPbin/Scripts _ X File Edit View Search Terminal Help	Status Stopped End On Stop
IOC: Unavailable Message: Identical process variable names on multiple servers Context: Channel: 'dp Linkaw,rxd5:ci94:dsc', Connecting to: 164.54.101.45:506 Requested Toyne: TVPENDTCONN Requested Count: 0 Source File:/cac.cpp Line number: 1297	Temperature 1
Thu Jun 27 15:23:59 CDT 2013 mednGAExceptionHandlerCh: Channel Access Exception: Channel New: Unavailable Native Court: 0 Native Court: 0 Access: Unavailable IOC: Unavailable	Pace
Message: Identical process variable names on multiple servers Context: Chambel: "dp Linkaw,rdd5:c149:cca01s", Connecting to: xraydetector4 5.xray.aps.anl.gov:5064, Ignored: 169.254.1.1:5064 Requested Type: TYPENDTCON Requested Count: 0 Source File:/cac.top Line number: 1207	$\underbrace{10_{+0}}_{0,0} \underbrace{-10_{+}0}_{-0,0} \underbrace{-5_{+}0}_{-0,0} \underbrace{-7_{+}0}_{-0,0} \underbrace{-5_{+}0}_{\text{Time}} \underbrace{-5_{+}0}_{-5_{+}0} \underbrace{-5_{+}0}_{-2_{+}0} \underbrace{-1_{+}0}_{-1,0} \underbrace{0}_{+,0}$

- The following parameters can be set (left side):
 - Pump speed for LN2 cooling (optional—this can also be controlled automatically based on the selected cooling rate). Upper limit = 30
 - Rate of temperature change (°C/min)
 - Desired temperature
- Note the control buttons (right side):
 - Scan Disable: tells VME (EPICS sscan) to stop communicating with the controller (e.g. software is loaded but controller is not in use). "Enable" restarts communication.
 - Toggle LNP between automatic & manual
 - Start, hold, or stop heating/cooling



EPICS Software Controls



Thanks to John Hammonds for the EPICS interface!

Appendix: Linksys Software Controls

- The Linkam equipment can be controlled with either EPICS or vendor software called Linksys, but not both simultaneously.
 - The Detector Pool no longer supports the Linksys software, but some beamlines may have it installed on their computers.
 - To use the Linkam software, make sure that the EPICS IOC is not running.
- From the desktop, click Linksys32 icon

Select "File: Connect"



Start and stop temperature ramps		X Linksys32 : Connected to > THMS 600 Stage File Setur View Video Help		
	1	^A ⊆ ∠ ∠ ↓ ↑ ⊕ ▶ ■ ∞ ^A ^A ∠ ↓ ↑ ⊕ ▶ ■ ∞ ^A	Da	ata capture
Set the rate, max/min temperature, and		Rate*C/min Limit*C Hold mins Lnp Delaysecs		controls
hold time here		User : linkam		



Appendix: Linksys Software Controls

	Linksys32 : Connected to > THMS 600 Stage	
	File Setup View Video Help	
Select "View	A Temperature Profile 🕨 🔳 👘	
Profile" to setup	27.: Real Time Chart ished Stopped	
multiple-step temperature ramps	Rate*C/min Limit*C Hold mins Lnp Delaysecs	
	User: linkam	

A previously-set temperature profile may affect your ability to control the stage. If control difficulties occur, check the temperature profile settings.

Profile						
	Profile - Cycle mode off					
Ramp	Rate	Limit	Time	Delay		
1	1	20.0	30	-		
2	0	0.0	0	-		
3	0	0.0	0	-		
4	0	0.0	0	-		
5	0	0.0	0	-		
6	0	0.0	0	-		
7	0	0.0	0	-		
8	0	0.0	0	-		
9	0	0.0	0	-		
10	0	0.0	0	-		
	0	مما	0			

If you are using the DSC, you may be required to use a Temperature Profile!!!