

I. The Quick-Step Guide

X-ray system users must be listed on the Radiation Safety **AUTHORIZED USERS** list in order to use the equipment. No one else is authorized to use the instrumentation. Users must use the on-line scheduling calendar to use the instrument. (<http://xray.utmb.edu/xcal>)

START-UP

XRG

Set the X-ray generator (XRG) to 40/14, 40/16, 40/18, 40/20,24,26,28,30 (kV/mA) waiting 10-15 minutes at each step.

The maximum XRG setting is: **40 kV, 30 mA**

CRYOCOOLER

- Turn ON CRYOCOOLER
- Set LN₂ boil-off power to level 2
- Wait 10 minutes for nozzle temperature to reach ~ 100 K

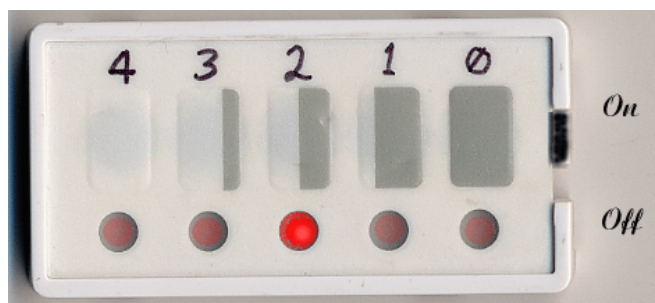


Illustration 1 Nitrogen Gas Generator Control (ON)

FINISHING UP

- Turn off the CRYOCOOLER
- Lower XRG current to 10 mA
- Keep XRG voltage at 40 kV

The stand-by XRG setting is: **40 kV, 10 mA**

NEVER lower XRG Voltage below 25 kV !

CHANGING LN2 TANKS

- Press the **{menu}** button on the LM-500 (This turns off the autofill)
- Change the LN₂ TANK on the refill line. (Tighten & loosen by hand. Only use the wrench to “seal” the connection ~1/8 turn)
- Press the **{menu}** button on the LM-500 so that the %LN₂ levels are displayed (IMPORTANT: This turns **ON** the autofill- The dewar will run dry if this is off!)

WARNING: DO NOT LET THE CRYOCOOLER RUN DRY - IT WILL BE DAMAGED

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1. XRG (X-ray generator) setting.

Set voltage/amperage (kV/mA) from: 25/10, 30/12, **40/10 (kV/mA, standby state)** - 40/14 - 40/16 - 40/18 - 40/20,22,24,26,28 - **40/30 (working state)**. Always increase voltage first and decrease amperage first. Wait at least 10 minutes at each step.

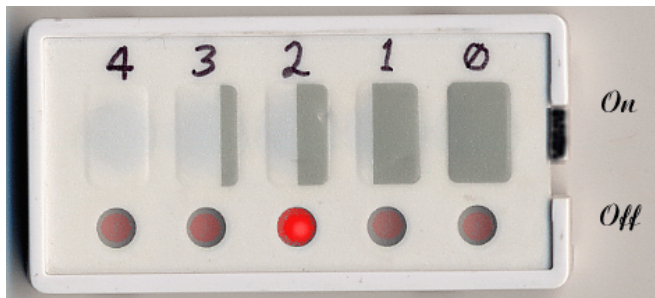


Illustration 2 Nitrogen Gas Generator Control (ON)

The Liquid Cryogen Level Monitor LM-500 detects the level of cryogen in blue dewar and an inside tank that directly supply cryogen to the system. The lower line of LM-500 indicates the level of blue dewar and the top line indicates the level of inside tank. The cryogen level in inside tank is automatically maintained between 80-85% and it is refilled from the blue dewar. If the cryogen level in blue dewar is under 40% (10% on CCD unit), it will be refilled from the transport dewar. If the LM-500 is showing an F or FA for a long period and is not refilling the blue dewar, the transport dewar needs to be changed. Also check the pressure gauge on the transport dewar, if it has dropped below ~40 psi then it is completely empty.

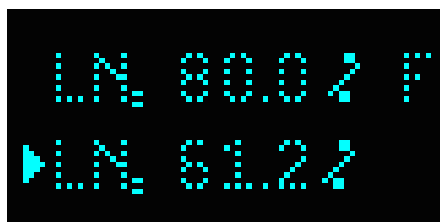


Illustration 3 LM-500 Auto refill display

(DO NOT LET THE CRYOCOOLER RUN DRY - IT WILL BE DAMAGED)

If there is no LN₂ available to refill the blue dewar turn off the Gas Generator. Always have a "FULL" dewar on the system overnight or on weekends. Empty partially filled dewars during the daytime by initiating a manual refill.

Normal Exchange of Transport Dewars

If the liquid level in the Blue dewar is > 40% then the transport dewar may be exchanged without the auto-refill venting the dewar. (Hitting {MENU} twice will turn off a fill cycle.)

Close valve on the Transport dewar

Use the adjustable wrench to loosen (½ turn) the brass compression nut on the refill line at the Transport dewar end.

Hold the refill line with the right hand while manually unscrewing the brass nut with the left.

Gently lower refill line to the floor.

Exchange transport dewars.

Hold the refill line onto the transport dewar's flair coupling with the right hand while gently tightening the brass nut by hand. If the nut cannot be fully tightened into place by hand do not proceed. The threads will be damaged by forcing the connection.

Use the adjustable wrench to seal the connection (½ turn).

2. The CRYOCOOLER.

Turn the LN₂ gas generator controller on and set at level 2. Wait about 10 minutes after which the the gas temperature should reach about 100K.

The Liquid Cryogen Level Monitor LM-500 detects the level of cryogen in blue dewar and an inside tank that directly supply cryogen to the system. The lower line of LM-500 indicates the level of blue dewar and the top line indicates the level of inside tank. The cryogen level in inside tank is automatically maintained between 80-85% and it is refilled from the blue dewar. If the cryogen level in blue dewar is under 40% (10% on CCD unit), it will be refilled from the transport dewar. If the LM-500 is showing an F or FA for a long period and is not refilling the blue dewar, the transport dewar needs to be changed. Also check the pressure gauge on the transport dewar, if it has dropped below ~40 psi then it is completely empty. The CRYOCOOLER cannot be operated without a transport dewar connected to the refill line. The nitrogen gas boil-off can vent out through the refill line instead of cooling the sample. The refill line will automatically vent at when the LN₂ level is below 40%.

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If the Auto-refill needs to be turned off while exchanging Transport Dewars

To change the transport dewar, first hit [MENU] button on the LM-500 once to turn OFF the auto-refill of both chambers, under this mode, the transport dewar and the blue tank is isolated - DO NOT LEAVE IN THIS STATE for more that a few minutes). Close the switch on the supply tank. Use a tool to loosen the screw and disassemble the tube by hand. Reconnect the transport dewar in reversed steps (open the valve half-way). Hit [MENU] button on the LM-500 again to return to the level display, turning on the autofill.

Turning off the auto-fill (temporarily)

[MENU] Turns off the refill on both the inner and outer dewars. If the unit is left in this state for long the liquid nitrogen in the inner dewar will be expended. Your sample will warm up and the nitrogen generator system may be damaged.

Manual Fill (navigating the LM-500 menu options)

[MENU] (with arrow on lower level display)

[V] FILL [ENTER]

[V] AUTO [ENTER] (displays START)

[MENU] [MENU] (return to main display. Outer dewar refill starts)

3. CRYSTAL MOUNTING.

Adjust the goniometerhead, using an empty cryo-pin of the correct size, so that the crystal will be cooled by the nitrogen gas stream. Move the detector back to 250mm so that you have room to work. Adjust PHI~30°, and the large arc Chi~60°. Put the cryo-pin holding your previously frozen sample on the goniometerhead and remove the storage tube **quickly** (otherwise, ice may form). Move Chi to your desired value (0° for most samples) and roughly center the loop. Tighten the screw on the Chi-slide so that the cryo-loop cannot move. Using the goniometer key adjust the goniometer head to center the crystal, at Phi=0, +/-90° and 180° [BASIC OPERATIONS/Goniometer control PHI]. Set the image plate to desired distance. A Resolution chart is posted on the side of the XRG.

4. DATA COLLECTION. (Always sign-in on-line & then fill-out log-book)

Login DIP2030 (ID 2030user, password *****).

There are three "Desktops" set-up to provide users with adequate working space

XPRESS This is for the data collection software "*xpress*", do not use for anything else.

DENZO Use this desktop for looking at your images with "*dip*" (xdisp), and processing with denzo and scalepack (HKL).

SCALEPACK When another user is using the DENZO desktop and you need access to files use this desktop instead.

XPRESS – This program should never be closed! If it is first type "*dipclean*" in a shell window then run "*xpress*". Minimize this window so no one is tempted to use it.

The first time XPRESS is run the Initialize/Manual and DIPmeasurement menus for unit A will need to be opened – Never close these windows.

Right-Click [Unit A] – [Initialize/Manual]

Right-Click [Unit A] – [Standard Measurement] – [DIP Measurement]

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Crystal Alignment requires that the PHI-axis be driven to +/- 90°, 0° (30° for cryo-mounting) from the **BASIC OPERATIONS** menu. Close this menu before starting data collection.

Set parameters of Goniometer Option in [Basic Operation] window and align the crystal. The crystal should be centered so that as phi rotates the center of the crystal does not move (Check at PHI = 0*, 90* and 180*).

For Safety Close the Shutter when handling the crystals. Before starting data collection, open X-ray Shutter (right). If the red light is off, it means the Shutter is closed, re-open it. And the X-ray status should be 'ready' (green indicator).

4.1 DIP Measurement

Choose a directory to save the images to. My working directory is:

```
/DIP1/data/{my_pi}/D{pg#}
```

(Note: It is a good idea to copy the DIP.DCL file from another directory into the current data directory)

```
cp D123/DIP.DCL D124/
```

Typical data collection parameters are:

Oscillation

Range:	0.5	Width of frame in degrees (for longer exposures reduce phi-range to 0.25 or less)
Interval:	0.5	Step between frames (normally = range)
Speed:	0.5	Deg/min (for 1min/osc set=range, longer exposures may use ½ range)
Repetitions:	11	#osc of phi range. Use an odd number so collection ends at phi-end, not phi-start.

Collect 1-4 frames of preliminary data to determine the appropriate parameters for data collection.

5. IMAGE PROCESSING.

Although this can be done on DIP2030, it is now recommended that all data be transferred to FENCHURCH for processing.

```
cp -puf *.ipf /data01/my_pi/D123
```

The image plates are recorded as *.ipf file (for example, D180_YedU_4_2nnn.ipf, datapage_crystal#_image#). Images can be viewed in DENZO or SCALEPACK desk

```
dip D180_YedU_4_2001.ipf
```

OR use HKL2000 to view and process your data.

Make a data processing directory, to keep the images separate from the processed data:

```
mkdir proc  
cd proc
```

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Copy the latest HKL denzo COM file to your proc directory (denzo-list will list old COM files)

```
cp ~/DENZOPARAMETERS/dz_firs_file_in_list.com dz_my_data_set_name.com
```

Edit the COM file using jot. Note that the common CTRL-* functions are accessed using the ALT-* key combinations(Cut @X, Copy @C, Paste @V, Save @S). CTRL-R does a search and replace (IRIX 5.3).

After the images are recorded, index one image first interactively by dz_D180_YedU_4_4.com. Examine the resulting log file and paste all the final parameters into dz_D180_YedU_4_4.com and rename as dz_D180_YedU_4_4b.com.

Comment out the [peaks file peaks.file] line, and autoindex will not be performed.

First determine the parameter. Run dz_D180_YedU_4_4b.com and all images will be refined and *.x file will be produced. Copy peaks.file as *.peak.

The *.x files will be processed by Scale_D180_4_#.com to get the space group.

Re-index by scale_cross.com

5.2 Processing offset (IP POSITION !=0) image data in HKL

<i>IP Position</i>	<i>X beam</i>	<i>Y beam</i>	<i>Cassette Rotz</i>
-30	50.0	123.2	60
-20	81.6	137.9	70
-10	115.3	147.0	80
0	150.0	150.0	90
10	184.7	147.0	100
20	218.4	137.9	110
30	250.0	123.2	120
X beam = $150 - 200 * \sin(\text{IP position})$			
Y beam = $150 - 200 * (1 - \cos(\text{IP position}))$			
Cassette Rotz = $90 - (\text{IP position})$			

5.3 Processing CCD data

Processing is best done on the machine hosting the data disks (Currently the Bruker2K Windows2000 machine). Use the KVM switch (2x **Scroll Lock**) to chose the correct machine to work on. The KVM switch has an auto scan feature ([L Shift], [R Shift]) to scan ([Space Bar]) to exit.

6. BACK-UP THE DATA (see the [Data archiving How-To](#))

First, move the image data to /data01/

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With 18Gb on DIP2030:/DIP1 and 500Gb on fenchurch:/data01 disks we can store about 3 months of data. Image files will be compressed after one-month and erased after 6 months on /DIP1. Data archiving is done on fenchurch using the **k3b** DVD+ writing software. Due to network incompatibility with data-collection, file transfers from DIP2030 to fenchurch should not be performed while images are being downloaded from the detector. A simple com script "denzo-save" will copy the denzo com files to /DIP1/data/DENZO_PARAMETERS, to keep an archive of data processing parameters. All data (image files *.ipf, *.x, .com and .log files) should be backed-up on DVD. first compress all images and remove all unwanted files and "*.bak".

processed files (*.x, .com and .log files) can be transferred to fenchurch or eddie and saved to CD/DVD.

Note that DIP2030 is now connected to fenchurch via a gigabit network connection. The /data01 disk on fenchurch is cross mounted on eddie, eniac, trillian and thrashbarg, where they can be processed directly, without any further transfer of files!

DONOT TRANSFER a LARGE VOLUME of FILES To/From the /DIP1 DISK While Collecting Data – Data loss will occur due to excess ethernet traffic. Use the “cp -pr * /data01/my_name/Dxxx/” command to transfer files after data collection has finished

7. The rotation instrument.

The crystal is rotated in small steps through the Ewald sphere. The spot positions are determined by the crystal orientation, the unit cell parameters, and crystal-to-film distance, the wavelength, and the film center. Data are collected in contiguous oscillation ranges, which is determined by the distance between the reciprocal lattice points (Unit cell parameters), the maximum angle of reflection (resolution), and the width of the spots.

8. TROUBLE SHOOTING

- XPRESS windows do not all appear:** Communications error - (1) Exit xpress (2) issue the dipclean command (3) Press the red RESET button on the DIP controller next to the XRG (4) start xpress and look for a long list of commands to be displayed in the shell -these are the software commands and replies to the DIP controller. Problem solved.
- TimeOut ERROR or Partial image:** Ethernet communications error – (1) Do not transfer files across the network (/data*) when collecting data.
- No X-rays:** Is the alarm light flashing? Then the filament needs to be replaced. A representative DIP image is shown at the right. Note the lack of a beam-stop shadow.

