Users Guide for HXMA Diffractometer Operation
Version 0.5

This document provides brief users guide for psi-8 diffractometer in HXMA.

HXMA beamline
Canadian Light Source Inc.
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1 Introduction of HXMA psi-8 diffractometer

HXMA psi-8 diffractometer has versatile configurations to accommodate various diffraction experiments. Available X-ray energy range is 5 keV – 40 keV.

1.1 Available equipments

Cyberstar scintillation detector
CCD X-ray detector
Vortex XRF detectors (four-element)
Closed loop cryostat (10 K – 450 K)
Analyzer crystals - HOPG(002), Si(111), Ge(111)

Figure 1.1
Diffractometer setup with CCD and Vortex detector

Figure 1.2
Psi-8 diffractometer detector arm with analyser setup.
1.2 Sample environments

1.2.1 Inert gas enclosure

To hold sample under inert gas (He or Ar) environment during X-ray scattering sample holder shown in figure can be used. Exposing sample to air during X-ray scattering measurement can build thin carbon layers on the sample surface. To prevent carbon buildup sample needs be placed under inert gas environment. There are two types of sample holder with sample surface either parallel or perpendicular to the phi stage. The sample holders have hole(s) for suction-holding of wafer samples. Also there are gas inlet and outlet.

![Figure 1.3](image)

Horizontal (left) and vertical (right) inert gas environment sample holders.

1.2.2 Beryllium mini-dome from ambient pressure to UHV

A sample sensitive to the air or moisture can be handled with UHV Beryllium dome. The vacuum pressure in Be-dome can reach high-vacuum (1e-6 Torr) with TMP. Bakeout of Be-dome is required to achieve UHV.
1.2.3 Low temperature with closed loop cryostat

Low temperature (10 – 450 K) sample environment can be achieved with closed loop cryostat.

Figure 1.5
Cryostat installed on psi-8 diffractometer. Beryllium radiation shield and shroud are removed.
1.2.4 High temperature

The mini Be-dome in combination with PBN heater sample can be heated up to 600 °C under ambient pressure and 800 °C in UHV.

1.2.5 Flow-cell/furnace type in situ reaction cell for powder diffraction

In-situ reaction cell has been provided to X-ray diffraction user community. The flow-cell/furnace design allows control of the sample temperature with possibility of reaching temperature of up to ~900 °C and also control of the atmosphere over the sample from rough vacuum to atmospheric flow to an overpressure.

![In-situ reaction cell mounted on psi-8 diffractometer with CCD detector.](image)

Figure 2. In-situ reaction cell mounted on psi-8 diffractometer with CCD detector.

1.3 Control

SPEC® has been used for diffractometer control, reciprocal scan, and data acquisition. Basic SPEC commands are described in separate document of “SPEC short manual for HXMA”.

1.3.1 Step Motor control

Step-motor control is based on EPICS control. Built-in EPICS motor control of SPEC is used through EPICS soft channels that bridges APS motor record and CLS motor record. This is because SPEC support EPICS motors based on APS motor record which is different from CLS motor record.
1.3.2 X-ray fluorescence detectors
Spectra from X-ray fluorescence detectors (single-, four-element Vortex®) are handled through mca EPICS control. Region of interests and other detector parameters are primarily set in mca EPICS. SPEC reads ROI setting from EPICS and also ROI’s can be set by SPEC. Mca spectra are saved in separate data files. Main SPEC data file records ROI’s as counters along with other parameters needed for dead-time correction.

1.3.3 CCD detector
SPEC scans with X-ray CCD detector (Mar165) has been provided by indirect control. Area detector EPICS control is used for CCD image acquisition. The CCD image can be saved to CCD control linux PC only. Through the EPICS control, SPEC set CCD image file name which reflect scan number and points in SPEC data file. SPEC also controls exposure time and put comments to CCD image file head. The comments include angle positions of diffractometer and incident X-ray energy. ROI’s within CCD can be set with EPICS or by SPEC. Background subtracted integrated intensities within ROI’s are recorded as counters in SPEC data file. CCD shutter control relies on time delay set by SPEC, and beamline fast shutter uses gate signal from scaler to control exposure time.
2 Where to start

2.1 Starting software SPEC

SPEC can be started from any Linux OPI computer at the beamline. It is recommended for user to make new folder for each beamtime (or each project).
Go to the folder where data will be saved and type
> runSPEC
The command will start SPEC for psic diffractometer.
If four-circle geometry is required, type
> runFOURC
Using psi-8 diffractometer as four-circle geometry means not including nu, mu angles are at their zero positions and not included in reciprocal space calculation.
Redefining standard macros can be possible through spec.mac. SPEC loads spec.mac in starting directory as last macro to read-in. Any standard macro redefined in spec.mac can be effective if SPEC starts from the directory containing spec.mac.
If macros are not work properly due to macros loaded by previous users, fresh start of SPEC is possible by typing
> runSPEC –f
For more option of starting SPEC type
> runSPEC --help

2.2 Pre-Checking

HXMA supports various experimental techniques which require quite different beamline configurations. Checklist for diffractometer can be found in Appendix.

2.2.1 Hardware limit switch

Hardware limit switch positions can be changed depending on experimental type. It is users’ responsibility to set hardware limit switch to protect diffractometer and equipments.
2.2.2 Disable unnecessary detectors

X-ray fluorescent detector and X-ray CCD detector need to be disabled if they are not used. Often these detectors were enabled from previous run. If EPICS for these detectors are not ready, SPEC can be crashed or hanged-up. If corresponding EPICS is running (started by previous users but not terminated) then there will be unnecessary waiting time in count or scanning.

```
> mca_off
> ccd_off
```

2.2.3 cyberstar window setting

Users need to properly set SCA window of scintillation detector (cyberstar, see 3.3). The window needs to be set according to the incidence X-ray energy. Also the window setting needs to be changed if gain or high voltage of detector is changed.

2.3 Sample alignment

Proper alignment of sample is crucial for diffraction measurement. Beamline provides motorized sample goniometer (X, Y, Z, θx, θy) for inert gas environment sample holders or capillary tube.

Closed loop cryostat alignment needs to be done manually.

2.4 MOSTAB

Most of anomalous scattering measurements (involving energy scan) Monochromator Stabilization (MOSTAB) is vital part to keep height of incidence X-ray constant. MOSTAB controls piezo for theta angle of monochromator second crystal based on the normalized difference of outputs from split ionization chamber or quadruple pin-diode BPM.
3 Using detectors

Pin diode, ionization chambers and cyberstar detectors are configured as default counters when spec starts.

3.1 Pin Diode (ip)

*ip*0: measure flux scattered off from Kapton film in the beampipe end.
It can be used to monitor flux incoming to the sOE hutch.

*ip*1: used for energy calibration.
Standard metal foil can be placed before the pin diode.

*ip*2: on detector arm filter box, used for bulk peak measurements.

3.2 Ionization Chambers (IC's)

*i*0: the first IC that monitors flux incoming to the defining slits.
*i*1: IC after beam defining slits, used to normalize signal (monitor counter).
*i*2: IC on detector arm.
Use *ic_info, ic_sens* and *ic_offset* to display and change gain and offset of SR570.

3.3 Scintillation detector (cyb)

It is cyberstar detector on the detector arm.
Use *cybermenu* to set up parameters for the cyberstar detector.
High voltage, gain, window range can be set through *cybermenu*.
*cyberscan* can be used to scan SCA window to select proper window and reject XRF and higher harmonics.

Known settings:

<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>Gain</th>
<th>Window low</th>
<th>Window high</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

12 | P a g e
3.4 Using X-ray fluorescence (XRF) detectors with spec

SPEC can take mca data if the EPICS control for MCA detector is up and running.

Typical step to follow:
Start EPICS control for mca detectors to use (see instructions of starting EPICS control below).
Start GUI for mca, get spectrum from standard or sample, and set ROI's.
To use four element Vortex detector (if it is not configured yet), use macro on_4ele.

`cf` In SPEC, use macros setup_mca for more flexible MCA setup.
Check ROI's with macros mca_rois.
Type `save_rois on`. Any scan will save ROI's to spec data file. Use `save_rois off` to disable ROI saving.
Type `MCAscanpt` before any scan macro, for example `MCAscanpt dscan eta -1 1 10 1`
To disable four-element Vortex use `off_4ele` and `save_rois off`.

Macros relevant to MCA setup and usage are:
ssetup_mca--> interface to configure multiple epics MCAs in spec.
show_mca --> display the configured epics MCAs nicely.
mca_rois --> to automatically check the created ROIS in each MCA and display their parameters.
save_rois -->to turn on/off the feature of saving MCA_ROIS(defined in roi_pv[], and filled when mca_rois is called) to spec data file.
rm_mca --> remove one or all configured MCAs,also remove all rois related to that MCA
rm_roi --> remove one/all configured MCA ROIS.Can also rmove all rois related to one MCA by calling rm_roi mca_nme
getandsave_mca--> to start counting and save MCAs and save their spectra in spec file.
MCAscanpt-->to save the MCA spectra in a separate file on each scan point of whatever scan( MCAscanpt anyscan macro)
MCAscanend--> to save the MCA spectra at the end of a scan (MCAscanend any scan macro)

EPIC control for the XRF detectors needs be started beforehand.
3.4.1 Starting EPICS control of four-element Vortex detector

Make sure IOC (computer) IOC1606-017 is turned on and connected to the network (10.106). If something doesn't seem to be working, see ???.

Connect all cables associated with the detector and electronics (see the detector related instructions in Hardware below)

3. Set up a video camera to watch the front panel of the XMAP crate
4. Turn on the XMAP electronics.

5. Double-click the "Start XMAP IOC" icon on the desktop, or type "runXMAPIOC". This should present a window with a numbered menu of detectors, one of which you can enter by name or by associated ID number.

Enter the detector (e.g. 4-Element or 1 for the 4-element Vortex detector).

You will be asked for a password. Use either the hxma account password. Leave the window open (closing it will shut down the control application)

6. On the video monitor, wait for the lights to stop flashing (it should settle into a 1Hz blink).

7. Start the control GUI by double clicking on the "XMAP4" or "XMAP1" desktop icon (see Software->GUI)

Open the "ADC Trace" window for each element: DXPs->DXP Control, then in the "DXP Control" window, More->ADC Trace

set the "us per bin" to 100

make sure you see a sawtooth pattern that resets about 3-5 times per second.

Open the Baseline Plots and make sure all are fairly symmetric and that they are centered very near zero

Back on the DXP Control window, make sure "Energy Peaking Time" is set appropriately.
3.5 Using X-ray CCD camera with spec

MarCCD saves images on local linux PC and it is not possible to save to serve driver. Still SPEC can “control” MarCCD through EPICS control. The “control” means file name of CCD image can be set by SPEC. Also SPEC can insert some information to head of CCD image file (16 bit tiff format).

3.5.1 Hardware and timing control

Current CCD image acquisition is based on scaler, fast shutter and time delay. Scaler output is hard wired to the uniblitz fast shutter so that fast shutter opens only when scaler is activated (counting). If CCD is enabled (ccd_on) SPEC takes extra step to give enough time for CCD readout. When count (ct or any scan) starts SPEC sends command to start CCD. Then, scaler is activated and faster shutter opens. After specified counting time set by SPEC is elapsed scaler deactivated and fast shutter closes. SPEC waits predefined delay time to move to next point of scan or print counting results.

<table>
<thead>
<tr>
<th>CCD starts</th>
<th>Scaler activated</th>
<th>Scaler deactivated</th>
<th>CCD ends</th>
<th>count ends</th>
</tr>
</thead>
<tbody>
<tr>
<td>-----------</td>
<td>------------------</td>
<td>--------------------</td>
<td>----------</td>
<td>------------</td>
</tr>
<tr>
<td>Fast shutter open</td>
<td></td>
<td>Fast shutter close</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.5.2 Prechecking

Following two steps are required/checked to use CCD through SPEC.

- “Remote Control” in data acquisition from marccd linux PC.
- Starting CCD EPICS IOC

Details can be found in 9.2 Quick Start

3.5.3 Enable/disable CCD

ccd_on will enable CCD image acquisition through spec macro ct.
ccdoff will disable CCD.

3.5.4 CCD setup

ccd_setup : select CCD to use (only one choice for now) and start setup.
1) CCD PV name \texttt{ccd1606-002:cam1}
2) Detect Dist.
3) CCD next file sequence
4) CCD on (1) / off (0)
5) CCD file on (1) / off (0)  
   \# Before start \texttt{ccdscan}, turn off CCD file.
6) CCD shutter delay time  \# Use default value
7) CCD read delay time  \# Use default value
8) CCD polling rate  \# Use default value
9) CCD filename
10) Fixed EPOCH # in Filename  \# Put 0 for filename without EPOCH
11) MONITOR counter number
12) CCD_Status counter number
13) The 6 motors to record
14) Using timingbox or not  \# Use default value
15) Add additional log file
16) CCD verbose mode on
17) CCD ROI MinX  
   \# ROI rectangular box with corners of  
   \( (\text{MinX}, \text{MinY}) \) and  \( (\text{MinX}+\text{SizeX}, \text{MinY}+\text{SizeY}) \)
18) CCD ROI MinY
19) CCD ROI SizeX
20) CCD ROI SizeY
21) CCD ROI expansion in X  \# 21, 22 for ROI background estimation
22) CCD ROI expansion in Y
23) CCD Binning X  \# Use default value
24) CCD Binning Y  \# Use default value

\textbf{3.5.5 Scan with CCD}

\texttt{scancdd [any scan macro of SPEC]}

Perform any scan macro with CCD images saved at each scan point.

CCD files will be saved as

\texttt{CCD\_filename.scan\_number.point\_number.file\_sequence.tif}
For example,

```
scanccd dscan eta -10 10 100 2
```

will generate 101 CCD file with 2 sec exposure at each scan point. CCD filename might look like `ccdtest.S003.N0012.012.tif` for 12\textsuperscript{th} scan point if “9) CCD filename” was set as “ccdtest,” the scan is the third scan, and “3) CCD next file sequence” was set to 1 before scanccd starts.
3.6 Configuring additional detectors

It is assumed that the additional detectors are temporary in nature.

3.6.1 Adding IC to counters.

Use `u_column_add` macro which is based on `cdef` of `spec`.

The counter can be removed by `u_column_remove`.

#    `u_column_show`: display user defined data columns
#    `u_column_add`: add one new entry of user defined column
#    `u_column_remove`: delete one or( a set related to one key identifier)
#                           column/columns
#    `u_column_clear`: clear all user defined columns

3.6.2 Adding cyberstar detectors

Default cyberstar detector (`cyb`) is configured in counter setting.

This is to add extra cyberstar detector(s), which will be used for non-permanent basis.

Use `u_column_add` and

then use `cyberunitsetup`

Example) `cyberunitsetup 1 0 1 3 2 4 10 5 0.1`

This defines the second cyberstar unit as an SCA (type 0) model PPU5CH (model 1),
configured with address 3, on SPECs serial device 2 with 4 seconds timeout. The unit has
5 channels, from 10 to 14. The default window size for scanning is 0.1 V. This means
that scans on the motor cyb will scan the lower threshold and adjust the upper threshold
to give a window of 0.1 V.

Then use `cybermenu`.
4 Energy macros and scans

HXMA uses two-crystal monochromator with fixed exit height. The second crystal of the monochromator has three motors (not the same as three-motor mono available in SPEC); rotation, translations along the downstream and vertical offset. Monochromator is somewhat similar to four-motor (monu, mond, montrav, and monoff) monochromator in SPEC classification with some differences.

Monochromator energy is changed through EPICS control. Limits of X-ray energy is 5 keV to 24 keV for Si(111) mono-crystal and 7 keV to 40 keV for Si(220) crystal. High energy cut-off by focusing mirror is about 30 keV. X-ray energy between 30 keV and 40 keV is available without focusing.

Several energy changing macros are available depending on a tracking of second monochromator crystal positions.

\( mve \) changes energy with two motors of the second crystal are changed to properly track X-ray diffracted from the first crystal and maintain fixed exit height.

\( amve \) changes energy (\( mve \)) and moves analyzer theta and two-theta to corresponding Bragg condition of analyzer crystal.

\( FHmve \) changes energy with only vertical offset of the second crystal is changed to maintain fixed exit height. Since horizontal position of the second crystal is not changed scan energy range should be limited less than 2 keV.

5 Using analyzer crystal

Analyzer crystals are typically used to reduce background count especially to cut florescence (inelastic) X-ray from sample.

Also, using analyzer crystal is equivalent to use very tight slit for a good angular resolution. Although moderate angular resolution can be achieved with Soller slit, Soller slit is not implemented for the moment.

Available analyzer crystals are pyrolytic graphite(002), Ge(111), Si(111), and Si(004).
<table>
<thead>
<tr>
<th>Analyzer crystal</th>
<th>d-space (Å)</th>
<th>Bragg angle degree 10 keV</th>
<th>width μrad @10 keV</th>
<th>width degree @10 keV</th>
<th>Reflectivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>PG(002)</td>
<td>3.355</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Ge(111)</td>
<td>3.2662725</td>
<td>10.9409</td>
<td>58.939</td>
<td>0.00338</td>
<td></td>
</tr>
<tr>
<td>Si(111)</td>
<td>3.1354161</td>
<td>11.4035</td>
<td>27.597</td>
<td>0.00158</td>
<td></td>
</tr>
<tr>
<td>Si(004)</td>
<td>1.357675</td>
<td>27.1685</td>
<td>13.343</td>
<td>0.000764</td>
<td></td>
</tr>
</tbody>
</table>

5.1 **Aligning analyzer crystal**

It should be done after psi-8 diffractometer is aligned (See 7.1 **diffractometer alignment procedure**).

Make sure slit in the detector arm defines zero beam, that is $\delta \ell = m \mu = 0$.

Move analyzer two theta ($\alpha \theta$) to zero and open s2 silts ($s2hgap$ and $s2hgap$) wide enough.

Move analyzer crystal from beam path (for example $mvr az -5$).

Find straight through beam with analyzer two theta scan.

\textit{dscan} \ \alpha \theta \ -1 \ 1 \ 30 \ .3

Move to peak position and define $\alpha \theta$ as zero (the peak position should be close to zero).

Bring in analyzer crystal to beam path and cuts beam to half by repeating

\textit{dscan} \ \alpha z \ -1 \ 1 \ 20 \ .3

\textit{dscan} \ \alpha \theta \ -1 \ 1 \ 30 \ .3

5.2 **Analyzer macros**

\texttt{sa} \ \texttt{analyzer\_crystal}

Setting the analyzer to use. It will list the default analyzers and ask the user to choose one of them (by name). The user can enter a custom name and d-constant of a crystal of their own choosing. Use variable \texttt{d\_ana d\_ana\_n}
\textit{amve energy}

Change energy to energy and move \textit{ath} and \textit{atth} to corresponding Bragg condition of analyzer crystal.

\textit{aEscan} start end step dwell\_time [fixQ]

Escan with proper analyzer tracking. At each energy ath and atth move to satisfy Bragg condition of analyzer crystal.
6 Miscellaneous Hardwares

6.1 XIA filter box

Standard configuration for XIA filter boxes is two XIA filter boxes before sample (and monitor counter) and one after sample (before analyzer crystal). The first two filters can be mainly used to reduce flux to sample so that radiation damage to the sample can be minimized. The filter after sample is to protect cyberstar detector.

6.1.1 Control of the XIA filter box

The XIA filter box is controlled through “S2E1606-001.cs.clsie.ca” port 4. The address of first filter box is set to 0.

Example of spec command:

1774.PSIC> sockPut("s2e1606-001:4004", ",!PFCU00 S\r\n")
1774.PSIC> p sockGet("s2e1606-001:4004")

%PFCU00 OK 0101 DONE;
%PFCU00 OK PFCU v1.0 (c) XIA 1999 All Rights Reserved

CHANNEL IN/OUT (FPanel TTL RS232) Shorted? Open?
  1  OUT OUT OUT OUT NO NO
  2  IN OUT OUT IN NO NO
  3  OUT OUT OUT OUT NO NO
  4  IN OUT OUT IN NO NO

RS232 Control Enabled: YES
RS232 Control Only: NO
Shutter Mode Enabled: NO
Exposure Decimation: 1
DONE;
### Table 3: PFCU command summary

<table>
<thead>
<tr>
<th>Command</th>
<th>Description</th>
<th>Arguments</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>PF2S2 Mode Enable shutter commands</td>
<td>None</td>
</tr>
<tr>
<td>4</td>
<td>PF4 Mode Disable shutter commands (initial default)</td>
<td>None</td>
</tr>
<tr>
<td>C</td>
<td>Close shutter</td>
<td>None</td>
</tr>
<tr>
<td>D</td>
<td>Exposure Decimation</td>
<td>Decimation value (1 to 65535)</td>
</tr>
<tr>
<td>E</td>
<td>Exposure</td>
<td>Exposure Time / (Decimation*10 ms) (1 to 65535)</td>
</tr>
<tr>
<td>F</td>
<td>Fault Status</td>
<td>None</td>
</tr>
<tr>
<td>H</td>
<td>Shutter Status</td>
<td>None</td>
</tr>
<tr>
<td>I</td>
<td>Insert Filter</td>
<td>Up to 4 filter numbers 1, 2, 3 or 4 (in any order)</td>
</tr>
<tr>
<td>L</td>
<td>Lock: ignore non-RS232 controls</td>
<td>None</td>
</tr>
<tr>
<td>O</td>
<td>Open shutter</td>
<td>None</td>
</tr>
<tr>
<td>P</td>
<td>Position Inquiry</td>
<td>Control Source (RS232, Panel, TTL) (optional – no argument gives overall setting)</td>
</tr>
<tr>
<td>R</td>
<td>Remove Filter</td>
<td>Up to 4 filter numbers 1, 2, 3 or 4 (in any order)</td>
</tr>
<tr>
<td>S</td>
<td>Status Display</td>
<td>None</td>
</tr>
<tr>
<td>U</td>
<td>Unlock: enable non-RS232 Control</td>
<td>None</td>
</tr>
<tr>
<td>W</td>
<td>Write Filter Position</td>
<td>0 or 1 for each filter</td>
</tr>
<tr>
<td>Z</td>
<td>Clear Short Error condition</td>
<td>None</td>
</tr>
</tbody>
</table>

### 6.1.2 Macros for XIA filter box

**fb_setup** [counter-mne] [min-count]

- `counter-mne` is the mnemonic of the counter to be used
- `min-count` is the minimum acceptable count for the counter specified. If the user enters a negative number, it will be interpreted as a percentage of the counter’s current count

**fbc**

this will update the coefficient matrix for the current energy

**fbu**
this will provide the log10 of the attenuation coefficient for the current filterbox combination, if an argument is provided then it will display the attenuation coefficient for the combination provided

\textit{fb\_on}

this will enable the automatic filterbox configuration during scans

\textit{fb\_auto}

this finds a suitable filterbox combination to bring the counts to the threshold
Slit control

There are three slits directly related to the psi8-diffractometer: beam defining slit, guide slit, and scattering slit. Currently XIA slits are used for all three slits. Beam defining slit is located before sample and normally monitor counter is located right after the slit. Guide slit and scattering slits are located after sample. The slits are referred as horizontal and vertical even though it is not quite correct for psi-diffractometer. Horizontal is along gamma direction and vertical is along delta direction.

6.1.3 Slit setting

Please note the quote from E. Vlieg for surface diffraction, “Most of the time, the detector arm will have two sets of slits: one set close to the sample (slit1) and one in front of the detector (slit2). In the in-plane direction, slit1 and slit2 together define the active sample area. The value of slit1 is the most important one for the active area, while slit2 is more important for the angular acceptance. In order to minimise the effects of the coupling between real and angular space, it is a good idea to have slit2 slightly larger than slit1. Typical values are 2 mm for slit1, and 3 mm for slit2. In the out-of-plane direction (l-direction), only slit2 is important. Slit1 (if implemented) should be kept wide open in that direction.”

6.1.4 Macros for slit

[under construction]
7 Data reduction and analysis program

7.1 Measurement from point detector

To extract structure factor from experimental data, several corrections should be applied. For scattering intensities measured through rocking scan with point detector, typical steps to pull out structure factors are; following

- Obtain integrated intensity from a rocking scan.
- Apply corrections of Lorentz-Polarization factor, area, and truncation rod acceptance length to the integrated intensity
- Average over symmetry equivalent rods

Users can use ANA-ROD program (http://www.esrf.eu/computing/scientific/joint_projects/ANA-ROD/) for data reduction and/or CTR analysis.

HXMA provides data reduction programs (made with awk language and CPLOT) for integrating intensities and applying corrections.

offspec.awk
correct_phi.awk (correction for nonspecular rod)
correct_eta.awk (correction for specular rod)

After averaged over symmetry equivalent rods, other programs (for example, ROD) can be used for structure analysis. Users also can use their own CPLOT programming for non-linear fit (*.5.c).
8 Diffractometer Alignment Procedure

Purpose: To make incident X-ray passes the diffractometer center AND \( \eta, \delta, \mu, \) and \( \nu \) base plates parallel to the X-ray at their zero angles (in user unit).

After step 8.1, 8.2, 8.3 \textit{align\_diff} macro can be used for automatic diffractometer alignment. Please refer step 8.4, 8.5, 8.6 for manual alignment.

See also step-by-step guide, CLS HXMA Psi-8 Alignment Procedure.

8.1 Mounting centering tool

REMOVE VORTEX AND ANY OTHER PROBES OR DETECTORS. MOTION OF THE CENTERING TOOL CAN HIT STUFFS AROUND SAMPLE/ SAMPLE STAGE. Centering tool is one directional slit mounted on extending structure from the \( \phi \) stage. The mounting does not require removing sample goniometer. But sample height \( \text{samz} \) need to be lowered to clear the X-ray pass.

8.2 Pre-checking of motor dials

If this is not a minor correction, motor dial positions must be checked before the align procedure starts.

For \( \nu, \mu, \eta \): \texttt{mvd motor 0} will move specified motor to zero angle in dial.

For \( \chi \): \texttt{mvd chi 90} will move chi to 90 degree.

Check \( \nu, \mu, \eta, \delta \) and \( \chi \) positions.

If the angle position is not zero or 90 (including dials in motor and reducing gear), turn motor axis to bring every dial to zero.

In stationary mode, power of a diffractometer motor is turned off automatically.

If you manually turned the motor axis, please record the amount in diffractometer log book. The record can be help beamline scientist to adjust maximum motor speed which does not lose step.
8.3 **Stationary ion chamber setup**

Place ion chamber on microprobe table and adjust its position so that the direct beam can get into ion chamber.

Adjust ion chamber gain properly.

8.4 **Diffractometer vertical height alignment**

Install centering tool on the \( \phi \) stage.

Set ion chamber on microprobe table (usually it is prekb) as detector (i.e. \( DET=prekb \)).

\[ \texttt{mvd chi 180} : \text{move chi to 180 degree in dial unit.} \]

Scan \( \phi \) to find peak. You find peak at \( \phi_0 \).

Then move \( \phi \) to \( \phi_0 + 180 \), and scan \( \phi \) to find peak. Let’s call this new peak position as \( \phi_1 \).

Now \( \phi_1 = \phi_0 + 180 \) then diffractometer has correct height. You can forward to horizontal position alignment.

If \( \phi_1 \neq \phi_0 + 180 \), then move \( \phi \) to \( (\phi_0 + \phi_1)/2 \) and do vertical table scan.

Find peak by vertical table scan:

\[ \texttt{d2scan vtbu start_rel end_rel vtbd start_rel end_rel step time}. \]

for example) \[ \texttt{d2scan vtbu 0 50 vtbd 0 50 50 1} \]

Raise or lower table to peak position. Now the X-ray is passing diffractometer center.

Forward to horizontal position alignment section.

8.5 **Diffractometer horizontal position alignment**

\[ \texttt{mvd chi 90} : \text{move chi to 90 degree in dial unit.} \]

Scan \( \phi \) to find peak. You find peak at \( \phi_0 \).

Then move \( \phi \) to \( \phi_0 + 180 \), and scan \( \phi \) to find peak. Let’s call this new peak position as \( \phi_1 \).

If \( \phi_1 = \phi_0 + 180 \) then diffractometer has correct height. You can forward to horizontal position alignment.

If \( \phi_1 \neq \phi_0 + 180 \), then move \( \phi \) to \( (\phi_0 + \phi_1)/2 \) and do vertical table scan.
Find peak by vertical table scan:

\texttt{d2scan} htbu \texttt{start\_rel} \texttt{end\_rel} \texttt{htbd} \texttt{start\_rel} \texttt{end\_rel} \texttt{step} \texttt{time}.

for example) \texttt{d2scan} \texttt{htbu} 0 5 \texttt{htbd} 0 5 50 1

Move table to peak position. Now the X-ray is passing diffractometer center. Forward to diffractometer table tilting adjustment section..

\section*{8.6 Diffractometer table tilting adjustment}

Now X-ray passes diffractometer center. However the incident X-ray might not parallel to rotation base of \textit{nu/mu} or \textit{del/eta} at their zero position.

We use different approach for yaw (\textit{nu/mu}) and pitch (\textit{del/eta}) adjustment. For yaw (\textit{nu/mu}), user unit of angles can be set to zeros instead of table rotation. But it is based on assumption that you did \textbf{Pre-checking of motor dials} (section 2.2) properly.

After diffractometer centering, move \texttt{phi} by 45° to clear beam path and scan \textit{nu} to find peak (with \texttt{prekb}).

Then move \textit{nu} to the peak angle, say \(v_0\).

Now set \textit{nu} to zero, by typing \texttt{set nu 0}.

It makes peak-position in user unit zero and dial unit is still \(v_0\).

Now move \textit{mu} to \(v_0\) and set to 0.

\texttt{mvd mu v0}; \texttt{set mu 0}

User unit of \textit{nu} and \textit{mu} are set to zero at peak position.

For \textit{del/eta}, table pitch (vertical tilting) must be adjusted.
9 Epics control of MarCCD (from HXMA wiki page)

9.1 Introduction

These are the configuration, startup, and basic usage notes for the MarCCD EPICS control software. The software is based on the synApps 'areaDetector' application. This control application connects directly to the MarCCD application and can write image files in TIFF format as per user specification.

These instructions can be found at http://wiki/index.php/HXMA_MarCCD_Detector.

9.2 Quick Start

On the MarCCD workstation, log in as user 'marccd'.

Double-click on the "MarCCD" icon.

On the MarCCD GUI, select Acquire->Remote Control...

On the Remote Control window, confirm or set the following

Server Environment: NETHOST=localhost

Server Command: /home/marccd/contrib/marccd_server/marccd_server_socket

Server Arguments: 2222

Server Log: /home/marccd/log/remote_server.log

Press Start on the Remote Control window, then Dismiss the window.

Go back to the data acquisition workstation

Double-click on the "MarCCD_IOC" icon.

Double-click on the MarCCD_GUI icon.

Select marCCD Drivers->marCCD #1 General, or change settings with marCCD Drivers->marCCD #1 Specific.

Start taking data.
9.3 Usage

Most of the MarCCD EPICS control GUI is informational. The following are the more commonly used controls.

9.3.1 Image Files

The image files are only available in TIFF format, using 16-bits per channel (i.e. 48-bit color/grayscale). The File format (not to be confused with Filename format) will display "netCDF" and should be ignored.

Note: due to the EPICS version of the controls being named similarly as the vendor's software, there can be some confusion as to whether the EPICS software can write image files. It doesn't do this. It can READ the image files written by the vendor's software for analytical purposes, but it is not set up to re-write it.

9.3.2 Single Shot Exposure

In the Area Detector screen, select marCCD Drivers->marCCD #1 General. That should launch the general purpose marCCD Detector Control window. Alternatively, you can launch the expert controls with marCCD Drivers->marCCD #1 Specific, which puts most of the controls in a single view with extra status displays.

In the Collect section:
Set the Exposure time (in seconds)
Ensure the following:
Acquire period is set to 0
# images is set to 1
Image mode is set to "Single"
frame type is set to "Normal"
Overlap type is set to "Sequential"
Array Callbacks is set to "Enable"
In the Readout section:
Set Binning for each axis according to your requirements.
Ensure that Frame shift is set to 0
In the **File** section:

In the general control window, press the **Driver file I/O** button. That will launch the files control window. The expert window has the same controls built in.

Set **File path** to the directory you want all of your images saved to.

Set **File name** to the **root name** of the files you want your images saved to.

Set **Next file #** to the starting value you want your sequence of images numbered from.

Set **Auto increment** to "Yes"

Set **Filename format** to a string as follows:

To specify the directory and root part of the file, start with "\%s/%s".

To specify the file number, at least use "\%d".

You can specify the number of digits with a number between \% and \d. This really isn't useful unless you also specify zero padding, below.

You can specify zero padding by putting a zero in front of the number, e.g. "\%04d", which would result in files numbered "0001, 0002,... 0999, 1000".

Between the filename root and the file number, and after the file number, you can add just about any set of characters.

Since the files are in TIFF format, it is highly recommended that the Filename format end with "tif".

Examples: assuming you set the File path to /tmp, File name to test, and Next file # to 5:

for a file that you want to look like "/tmp/test-0005.tif", the Filename format should be "\%s/%s-%04d.tif"

for a file that you want to look like "/tmp/test05.tif", the Filename format should be "\%s/%s02d.tif"

Set **Auto save** to "Yes"

To actually generate an image, go to the **Collect** section and press **Start**.

The various displays in the **Status** section will inform you of the status of the exposure.

### 9.3.3 ROIs (Regions-of-Interest)

In the **Area Detector** screen, select **marCCD Plug-ins->ROI #1**. That should launch the **ROI1** window.

Set **Enable** to **Yes**
Set Callbacks block to Yes
You can examine individual ROI information and modify the ROI's settings with
Individual ROIs menu.
Set Use this ROI? to Yes
Make sure Data type is set to UInt16
Set Compute statistics to Yes
Set histogram? to Yes if you are interested in seeing the quasi-distribution of pixel
intensities. At first, set the parameters as follows:
Set Size to 65535.
Set Minimum to 0.
Set Maximum to 65535.
Set ROI start and ROI size as you need. It is highly recommended that ROI #1 be set to
(0,0) and (2048,2048) respectively, that you pay attention to the Statistics section,
particularly the Maximum reading for overflows.
For multiple ROI information in tabular format select from the Combined ROIs menu.

9.3.4 Multiple Frame Exposures
It might be useful to set Overlap mode to Overlap when doing multiple exposures in
succession. This allows an exposure to begin while the previous file is being written. If
you do use this feature, AND you do not need ROIs, it would be advisable to set Array
callbacks to Disable if Overlap mode is set to Overlap. Callbacks will block the
overlap feature. If you want reliable ROIs, you have no choice but to keep Array
callbacks enabled.

9.4 Start-Up
The details of starting up the MarCCD system can be found here.

9.4.1 Hardware
The hardware system is fairly complex. Setup, configuration, and operating instructions
should only be performed by experienced personnel, thus are beyond the scope of this
document. If the MarCCD workstation, the cryogenics system, or the detector is not powered up, inform the beamline staff.

Regarding the workstation, it is not integral to the operation of the detector hardware. It usually can be rebooted with little or no impact to the detector or cryogenics. **HOWEVER**, the workstation should **NOT** be unnecessarily rebooted. It is still possible to lock up the detector this way.

Finally, the workstation **IS** integral to data acquisition, since it acts as the data and control server for the EPICS control software.

### 9.4.2 Software

On the MarCCD workstation, log in as user 'marccd'.

Double-click on the "MarCCD" icon.

this should launch the MarCCD GUI and a terminal window

the GUI is what users would normally use locally, and can be monitored for remote usage

the terminal window reports the status of the system's hardware.

On the MarCCD GUI, select **Acquire->Remote Control...**

On the Remote Control window, confirm or set the following

- **Server Environment:** NETHOST=localhost
- **Server Command:** /home/marccd/contrib/marccd_server/marccd_server_socket
- **Server Arguments:** 2222
- **Server Log:** /home/marccd/log/remote_server.log

Press **Start** on the Remote Control window.

**Dismiss** the Remote Control window.

Go back to the data acquisition workstation

Double-click on the "runMarCCD_IOC" icon.

There should be a fair amount of text and should end with a prompt such as "ccd1606-002>".

Double-click on the MarCCD_GUI icon.

The EPICS control window should appear.
9.5 Shutdown

in the MarCCD IOC window, type exit
in the MarCCD GUI, select Acquire->Remote Control...
Press "Stop".
You will be prompted with "Do you want to abort data collection Immediately?"
Answer "No,..."
Dismiss the Remote Control window
shut down the MarCCD EPICS control GUI (NOT the main MarCCD control GUI).
Don't forget to close the EPICS controls "main" window, also.
### 10 Appendix

#### 10.1 Table 1. List of spec motors

<table>
<thead>
<tr>
<th>spec name</th>
<th>Controller</th>
<th>spec conf.</th>
<th>description</th>
</tr>
</thead>
<tbody>
<tr>
<td>del</td>
<td>EPICS</td>
<td>psic</td>
<td>I1039</td>
</tr>
<tr>
<td>eta</td>
<td>EPICS</td>
<td>psic</td>
<td>I1040</td>
</tr>
<tr>
<td>chi</td>
<td>EPICS</td>
<td>psic</td>
<td>I1041</td>
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<td>EPICS</td>
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<td>I1038</td>
</tr>
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<td>psic</td>
<td>I1037</td>
</tr>
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<td>psic</td>
<td>I1035</td>
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<td>psic</td>
<td>I1036</td>
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<td>EPICS</td>
<td>psic</td>
<td>I11090</td>
</tr>
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<td>I11091</td>
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<td>tune</td>
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<td>common</td>
<td></td>
</tr>
<tr>
<td>ev</td>
<td></td>
<td>common</td>
<td>Energy changeable with track-on</td>
</tr>
<tr>
<td>s0hgap</td>
<td>XIAHSC</td>
<td>common</td>
<td>408</td>
</tr>
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<td>XIAHSC</td>
<td>common</td>
<td></td>
</tr>
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<tr>
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<td>434</td>
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</table>
### Table 2. Motor parameters

<table>
<thead>
<tr>
<th>Motors</th>
<th>Resolution Step per degree/mm</th>
<th>Sign of user*dial</th>
<th>Steady-state speed [Hz]</th>
<th>Base speed [Hz]</th>
<th>Acceleration time [sec]</th>
<th>Backlash [steps]</th>
<th>High limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>s1vcen</td>
<td>XIAHSC</td>
<td>psic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>s2gap</td>
<td>XIAHSC</td>
<td>psic</td>
<td></td>
<td></td>
<td>409</td>
<td></td>
<td></td>
</tr>
<tr>
<td>s2hcen</td>
<td>XIAHSC</td>
<td>psic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>s2vgap</td>
<td>XIAHSC</td>
<td>psic</td>
<td></td>
<td></td>
<td>410</td>
<td></td>
<td></td>
</tr>
<tr>
<td>s2vcen</td>
<td>XIAHSC</td>
<td>psic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- **del**: 8000 1 8000 800 250 200
- **eta**: 8000 1 8000 800 250 200
- **chi**: -8000 1 8000 400 250 200
- **phi**: -4000 1 16000 800 250 200
- **mu**: -8000 1 2000 200 125 200
- **nu**: -8000 1 2000 200 125 200
- **htbu**: 800 1 50 20 125 50
- **htbd**: 800 1 50 20 125 50
- **vtbu**: 16000 1 2000 2000 125 50
- **vtbd**: 16000 1 2000 2000 125 50
- **samx**: 200 1 200 200 125 50
- **samy**: 200 -1 200 200 125 200
- **samz**: 200 -1 200 200 125 50
- **ath**: 4000 1 8000 200 1000 100
- **athh**: -4000 1 8000 200 1000 100
### 10.3 Table 3. RS232 port assignment of Nport

Port assignment of 16-channel serial-to-Ethernet device “S2E1606-001.clsi.ca”

<table>
<thead>
<tr>
<th>Port number</th>
<th>Device</th>
<th>spec</th>
<th>Note</th>
</tr>
</thead>
</table>
| 1           | XIA slit                    | s1vgap, s1vpos  
s1hgap, s1hpos  
s2vgap, s2vpos  
s2hgap, s2hpos |                               |
| 2           | Cyberstar X2000 controller  | cyb         | counter                       |
| 3           |                             |             |                               |
| 4           | XIA S2F2 shutter            |             |                               |
| 5           | Lakeshore temperature       |             | EPICS control is optional.    |
|             | controller                  |             |                               |
| 6           |                             |             |                               |
| 7           |                             |             |                               |
| 8           |                             |             |                               |
| 9           | SR570                       | ic0         | counter                       |
| 10          | SR570                       | ic1         | counter                       |
| 11          | SR570                       | ic2         | counter                       |
| 12          |                             |             |                               |
| 13          |                             |             |                               |
| 14          |                             |             |                               |
| 15          | ESP 301                     |             |                               |
| 16          | Eurotherm 2416              | t_euro, europv | counter                     |