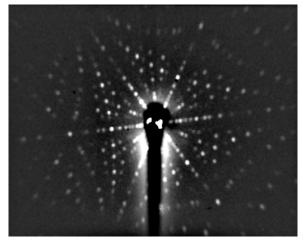


Orienting Crystals with the 1-CCD Laue Camera

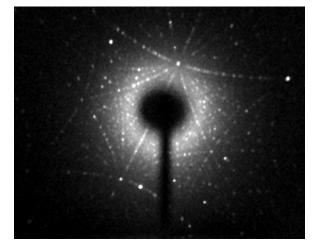


- Sensor Type: Sony ICX825ALA or ICX694ALG
- Image size: Diagonal 11mm (Type 2/3") or 16mm (type 1")
- Chip size: 8.98x6.71mm or 12.49x9.99mm
- Resolution: 1392x1040 or 2759 x 2200
- Pixel Size: 6.45x6.45 μM or 4.54 x 4.54 μM
- Binning: from 2x2 to 8x8 (improved intensity and read-out)
- High sensitivity: (QE>75% at 500-600nm), low smear
- Low dark current: 0.003@-10 °C, excellent anti-blooming
- Full well capacity: 20,000 or 40,000 electrons
- ADC: 16 bit grey scale image, optional filtering and distortion
- Readout Noise: 4 e- typical (slower readout = less noise)
- Readout Time: 1-3s
- Interface: USB 2.0 High Speed with 10-20m USB cables
- Power: 12v DC 0.8A, to EU, UK, US/Japan, AU/CN standards
- Maximum Exposure Length: Unlimited
- Minimum Exposure Length: 1/1000s (1s at full resolution)
- Cooling: Thermoelectric set point with max ΔT=-27°C
- CCD Unit: 60mm diameter, 122mm length, 400g weight
- Lens Coupling: T2-mount, C-mount, F-mount, micro 4/3
- SDK: C++, VB Wrapper, .net Wrapper, ImageJ, LabView

Backscattered Laue with ~300s exposures of the 1-CCD Laue Camera



a) Si (Dean Hudek, Brown University USA).



b) Sm2Fe17 (Léopold Diop T.U. Darmstadt)

Installing the NeutronOptics1-CCD Laue Camera on the x-ray beam

The photos below show how **Prof. W. Donner and Dr L. Diop at the T.U. Darmstadt** converted a Huber Image Plate Laue camera (left) to use the NeutronOptics 1-CCD Laue camera (right).





Left photo: The Huber IP camera is the circular disk mounted on a blue support on a guide rail. The collimator is locked and the beam shutter opened with the two black knobbed levers on the double-metal plates that form the <u>Huber-800</u> alignment mechanism, which is mounted on a 1960's Philips X-ray generator. The screw at the top, and two at the bottom, adjust the height and tilt of the collimator.

Right photo: The Huber-800 unit is retained, together with the blue camera support, when the Huber IP camera is replaced by the 1-CCD camera. An additional support bolt on the rail under the bottom left supports the heavier camera. The new collimator is aligned in the Huber-800 mechanism for maximum intensity, and the camera is then bolted on using an aluminium plate and four M4 bolts.

3mm Collimator-tube adapter



1mm Collimator nose

Mount the camera as close to the source as possible using the 4mm bolt holes at the back; minor adjustments to position and orientation are necessary. Before inserting the 1mm collimator tube into the camera, check that it fits the source exit and allows no x-ray leakage. You will need to make **a small adapter** (top left) to take the 2mm collimator tube, depending on your source. The 1mm collimator slides into the 2mm tube (bottom left), with the nose extending toward the crystal ~30mm from the window.

The carbon fibre window & scintillator are not fragile.

The camera and collimator must be precisely aligned, and the source voltage and current set for maximum x-ray intensity through the collimator. Our <u>mini i-Cam camera</u> could be used to align the beam and position the sample, otherwise use the Laue camera itself to check beam transmission.

Typical exposure times for backscattered Laue are ~300 seconds. But first try a shorter exposure with no beam to check that everything is working - you should see background noise.

Binning is important for faster exposures with backscattered Laue, since very high resolution is not needed. Typically binning of 4x4 increases efficiency by a factor of x16 ! You can also try forward scattered Laue, where exposure times are much shorter.

Installing the Driver and Artemis Capture software

- Browse the CD & launch "SetupArtemisUniversal.exe" or "artemisinstaller.exe"
- Install the driver & "Artemis Infinity" and "ArtemisCapture" to control the camera
- You can also download and pre-install the <u>Artemis core software & drivers</u>
- Plug the 12V supply into the camera; the fan should start up
- Connect the 10m USB extension to the computer, wait for its driver to be installed
- Then connect the camera to this cable, or directly to a USB port on your computer
- The computer should automatically search for and install the driver. Be patient !
- The Windows Device Manager should now show an ArtemisCCD USB controller
- Launch "Artemis Infinity" or "Artemis Capture" to control the camera (next section)
- Read the manual for the Hi-res camera for more details and troubleshooting

Artemis Capture for long exposures (~300s) in backscattering

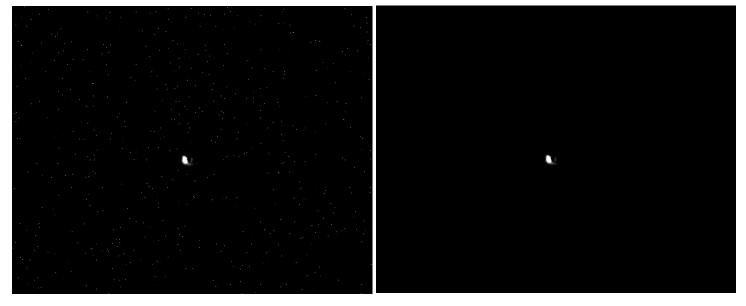
Launch Artemis Capture and "View" Exposure, Display & Cooler. The "File" menu is used to save images, the "Camera" menu to connect the camera, and the "Colour" menu should be set to "Raw".

Artemis Capture		
File Camera Colour Help		
		Exposure × Exp.(s) 30 min : sec. Dly.(s) 0 Pre BinX Pre BinX Pre BinX StartY Vidth 1391 Height 1039 Full frame Autosave Images Display × Black - Uvitte 5000 Zoom - Auto stretch Negative Cooler × Status: 0ff Temp: 26.9°C Cooler on -
 Image: A set of the set of the	III.	+
Ready	Not saved (78	3,1013)=304 Zoom 1:2 Exp. 14 sec

The camera icons launch a single exposure or continuous loop exposures. "Tracker", "Marker" and "Filter" icons are not used. **IMPORTANT: Zoom out to see the green outline of the full CCD.**

- Set exposure time with BinX=4 and BinY=X for 4x4 binning and Dly(s)=0 delay
- Check the "Auto stretch" option to select the best 8-bits out of the 16-bits.
- "Zoom" (or the wheel mouse button) zooms the display window
- Set the temperature to 5°C to reduce noise and use ImageJ Process/Noise/Despeckle
- If the CCD is cold, click "Warm up" and wait a few minutes before disconnecting power

Before mounting the camera, check it with no x-ray beam. The left hand image below is a raw 300s exposure with 4x4 binning and the scale adjusted to emphasise the background. Note that even with cooling, many isolated pixels are brighter than their surroundings; this is a normal variation in the manufacture of a highly sensitive CCD (click image to expand). Note also the bright spot in the centre of the image; this is due to residual light leakage around the collimator exit, under the rubber O-ring.

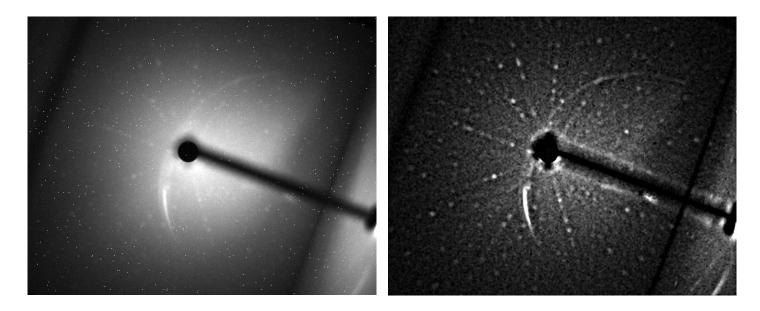


The isolated bright pixels can be filtered out with ImageJ's "Process/Noise/Despeckle" menu, which replaces the bright pixels by the average of their surroundings (right hand image). This will not filter out Laue diffraction spots, which are much broader. The central spot due to light leakage is not a practical problem, since it coincides with the incident beam. If necessary, it can be eliminated with a very little black paint under the collimator O-ring. Be careful not to allow paint to run through onto the scintillator, which is immediately behind the carbon fibre window. Or you can simply turn off the room lights when collecting x-ray images.

Emphasizing the Laue Pattern with imageJ

You can use ImageJ's **"Process/Noise/Despeckle"** to <u>filter out electronic noise</u> and **"Process/ Subtract Background"** with "rolling ball radius 25-50 to <u>remove some of the diffuse backround</u>. You might also try ImageJ's **"Process/FFT/Bandpass filter"** to filter out large structures of 30+ pixels and small structures of 3+ pixels, with "autoscale" and "saturate" to <u>emphasise the peaks</u>.

A more powerful macro routine can be <u>downloaded</u> to **ImageJ\macros\toolsets** then loaded by clicking on the **red** ">>" menu item on the far right and selecting "Subtract Diffuse Bkgd". This will install the command "**Plugins/Macros/Subtract Diffuse Bkgd**". The left image below shows a very poor image with isolated bright pixels and no obvious Laue pattern. The right image shows the astonishing effect of this macro command. This organic material showed strong diffuse scattering; hopefully your patterns will be better, but it is surprising that even very noisy images can yield useful patterns. This "median filtering" macro was provided by <u>M. Cammer</u> as an alternative to "**Process/FFT/Bandpass filter**". (click images to expand).



Hints on getting Optimal Performance

- Use a Windows-7 or -10 computer with 2GByte of memory eg an Intel NUC
- If necessary, close other applications and windows that may use memory
- Try a short exposure to check that it works before starting a long exposure
- Check the files that are created in your image directory
- Image display is for guidance. Open the saved image with ImageJ for measurement
- Note that the ImageJ lens distortion correction is not perfect far from the centre
- The CCD should be cooled to reduce noisy pixels. Also use ImageJ's despeckle filter
- Fogging may occur if you cool below 0°C (the CCD chamber contains a desiccant)
- The desiccant can be replaced by removing the large screw on the camera shaft
- The desiccant tablet can be renewed by heating to 100°C in an oven
- Try unplugging and re-plugging the USB cable or re-booting your Windows computer :-)
- The centre of the image doesn't correspond to the exact centre of the window

Indexing and orienting the Laue pattern

You may first want to use ImageJ's "*Process/Noise/Despeckle*" to <u>filter out electronic noise</u> and "**Process/Subtract Background**" with "rolling ball radius 25-50 to <u>remove some of the diffuse</u> <u>background</u>. After noise filtering, you might also try "**Process/FFT/Bandpass filter**" with "autoscale" and "saturate" to <u>emphasise the peaks</u>, before using imageJ to convert to the various formats used for indexing. A number of Laue indexing applications are freely available. The <u>Cologne Laue</u> <u>Indexing Program (CLIP)</u> is one of the best, along with the <u>QLaue Indexing Program</u>. Older applications such as <u>OrientExpress</u> and the ambitious new <u>ESMERALDA Laue suite</u> might also be tried. First try <u>WinLaue</u> to simulate Laue patterns and becoime familiar with what to expect.

For the latest information, please check our web site http://neutronoptics.com/laue-1CCD.html