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Large-Area CCD Detectors on Macromolecular Beamlines at CHESS

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Efficient collection of X-ray diffraction data from materials with large unit cells, such as protein crystals, requires an area detector which has a large area, high spatial resolution, good sensitivity to X-rays, and high precision and accuracy in recording intensities of diffraction spots. It is also highly desirable that it have a short readout time and be easy to use. At present, imaging plates and CCD detectors come closest to satisfying all these requirements. Both types can produce excellent data, but CCD's have the edge in speed of readout and ease of use. At CHESS, Fuji image plates are available, but most data are collected on CCD detectors.

CCD's at CHESS

CHESS has been involved in the development of CCD detectors for crystallography since 1993, when a 512 x 512 pixel detector developed by Sol Gruner's group (then at Princeton) was sent to Ithaca for testing. This was rapidly followed by a 1024 x 1024 pixel ("1K") Gruner detector, which stayed at CHESS on a long-term loan basis and was made available to users. The "1K" detector produced high quality crystallographic data (Thiel et al.,*Rev. Sci. Instrum.* **66**, 1477 (1995); Walter et al., Structure 3, 835 (1995).) and was a model later used by Area Detector Systems Corporation (ADSC) in producing its commercial detectors. In 1994 MacCHESS purchased a 2048 x 2048 pixel (80 mm. square) detector of the same type as the "1K" from Princeton Scientific Instruments; this "2K" detector has produced massive amounts of excellent quality data, was in constant use through 1998, and is still available as a backup detector. With the success of the CCD technology well established, in 1996 MacCHESS purchased its first commercial CCD detector from ADSC (an 1152 x 1152 pixel Quantum-1, serial number 001). Additional purchases followed, and all three of the macromolecular beamlines (A-1, F-1, and F-2) are currently equipped with ADSC Quantum-4 detectors.

The ADSC Quantum-4 detector

The Quantum-4 is a modular detector, comprising a 2 x 2 array of CCD chips. At the front of the detector a surface 188 mm square is coated with a phosphor which converts incoming X-rays to visible light. This light is transmitted through four tapered fiber-optic bundles to the four CCD chips. Thermoelectric coolers keep the chips at about -50 degrees C, and electronics at the back of the detector read them out. The chip/fiber-optic assemblies are in vacuum, but most of the electronics are, since a recent upgrade, outside the vacuum can, and hence easy to service. The complete system from ADSC includes one controller for each chip, a PC to communicate with the controllers, and data collection software to run on a workstation (which communicates with the PC over a local network connection).

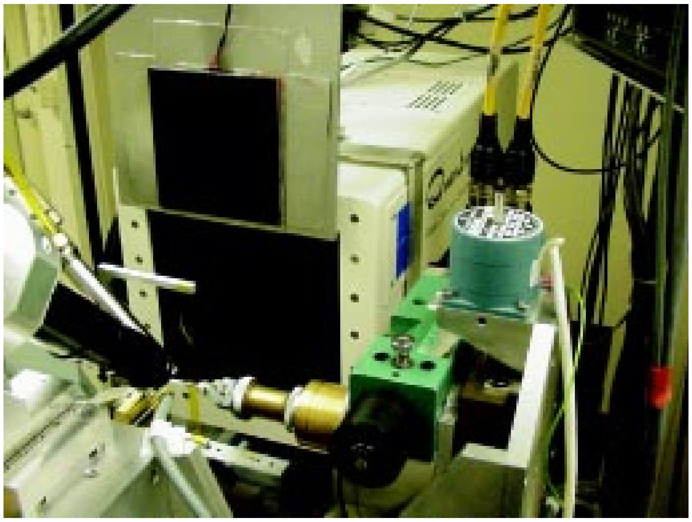
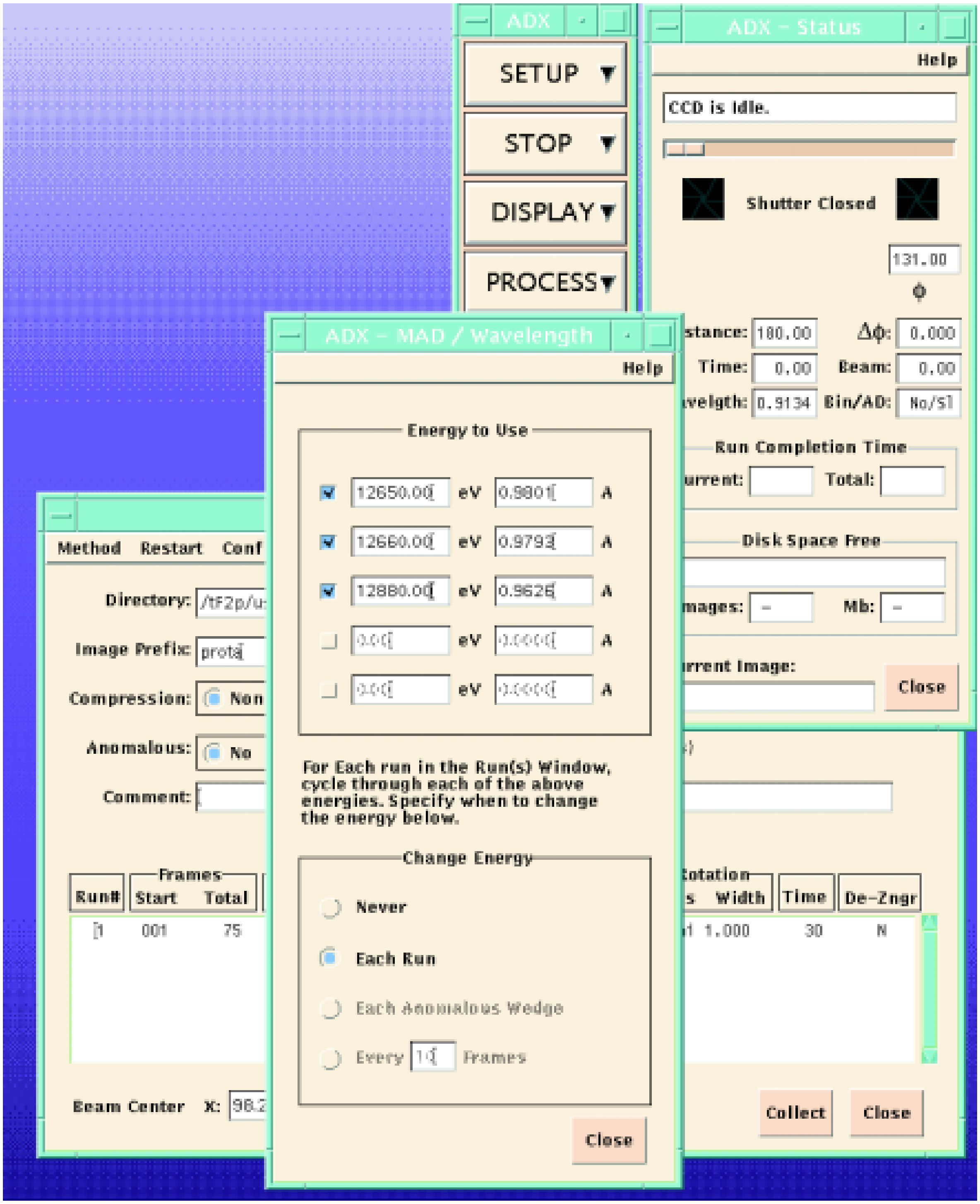


Figure 1: A view of the Quantum-4 detector installed at F-2, seen from the "upstream" end of the hutch. The rectangular object above the detector is a safety shield which protects the CCD from mechanical damage and from being struck by the direct X-ray beam. The shield closes when the hutch door is opened, and can only be raised when the door is closed and the beam stop is in place. The rotation stage for the crystal is at lower right, the cold stream nozzle at upper left, the crystal-viewing camera at center left, and the X-ray collimator at lower left.

The ADX data collection software

ADSC provides, with its detectors, a graphical user interface (GUI), called ADX, for collecting diffraction data by the oscillation method. This GUI is used to control station hardware such as X-ray shutters and goniostat motors, as well as the detector itself; this coordination is necessary (as well as convenient) for producing good data. The most recent version of the ADX software includes the ability to send commands to the beamline hardware to change the monochromator setting, and hence supports MAD (multi-wavelength anomalous diffraction) experiments. Some parts of the software require customization for each installation; MacCHESS staff members Jim LaIuppa and Marian Szebenyi have worked together with Chris Nielsen of ADSC to develop and maintain CHESS-specific code, as well as suggesting improvements to the interface in general.



CCD is Idle.

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☑	12660.00	eV	0.9793	A
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☐	0.00	eV	0.0000	A
☐	0.00	eV	0.0000	A

For Each run in the Run(s) Window, cycle through each of the above energies. Specify when to change the energy below.

○ Never
● Each Run
○ Each Anomalous Wedge
○ Every 10 Frames

Directory: /tF2p/u

Image Prefix: prota

Compression: ● Non

Anomalous: ● No

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Beam Center X: 98.2

Figure 2: Appearance of the ADX interface during setup for a MAD experiment. The window in front allows selection of up to 6 wavelengths at which to collect data. In back are the main control panel, a status panel, and a window in which to set per-run parameters such as oscillation width and number of frames to collect.

Processing data from the Q-4

The raw images from a Q-4 must be corrected for geometric distortions introduced by the fiber-optic tapers, and for intensity errors due to nonuniform response of the detector. These corrections are made automatically during data collection, using calibration files generated for each individual detector. These files are provided by ADSC with each detector, and are good for months or years. When recalibration is required, hardware and software for the purpose are available at CHESS. Corrected images are then fed in to a general oscillation processing package. A collaboration between Michael Rossmann's group at Purdue, MacCHESS, and ADSC is actively developing one such package, DPS, as well as a convenient graphical interface to run DPS programs, the integration program MOSFLM, and some CCP4 programs. All are available to CHESS users. The package may be downloaded from the Web; more information is available at http://www.chess.cornell.edu/MacCHESS/processing_gui.html. Data processing support is provided by Marian Szebenyi and Art Weaver.

Table 1: Some Quantum-4 specifications

No. of pixels: 1152 x 1152 per chip, 2304 x 2304 for full detector
Pixel size, 0.0816 x 0.0816 mm., bits per pixel:16
Gap between modules: 0.4 mm. (5 pixels)
Readout time: 1 sec (2x2 binned, fast) to 9 sec (full resolution, slow)
Dark current: 0.03 e^-/pixel/sec.

Future plans

Even larger area detectors than the Quantum-4 will be needed in the future due to recent achievements in growing crystals that contain large unit cells and also diffract to high resolution. Moreover, initiatives such as structural genomics will place emphasis on very rapid data collection and processing. To address these concerns, MacCHESS will continue to upgrade its CCD detectors. The new Quantum-210 detector, now being developed by ADSC, will have a 2 x 2 array of 2048 x 2048 pixel chips and it will have a reduced readout time. MacCHESS will be installing two of these detectors once they are available and tested. For even more area, Mike Cook has designed a mount for holding two detectors in a V configuration - this design allows collection of data to very high resolution without very oblique incidence of X-rays at high Bragg angles. Software improvements are planned to make on-line data processing easier - this will allow quick detection of problem data sets (which could perhaps be recollected if identified while users are still at CHESS), as well as a general speedup in structure solution.

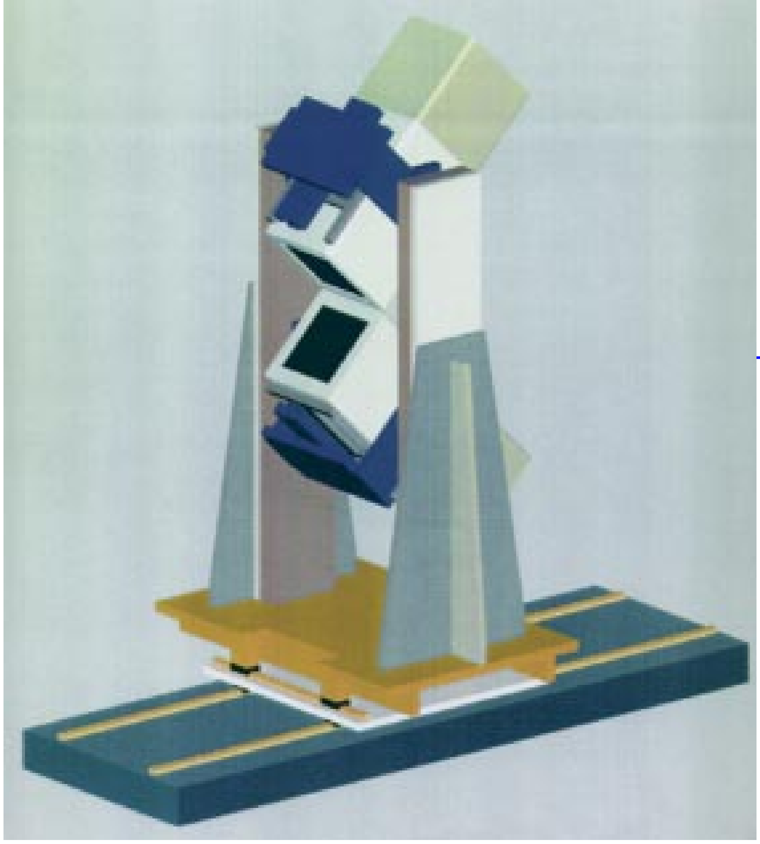

Figure 3: Design for a proposed dual-detector mount. The entire mount moves in all three directions, and the two detectors on the mount can be independently rotated and translated along their axes; possible configurations include one large flat area, one detector flat and the other tilted, and the V position shown.

Drawing by: *Mike Cook*