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# A new macromolecular oscillation camera at CHESS

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Increasing X-ray flux and decreasing crystal size are two factors placing new demands on macromolecular diffraction cameras at synchrotrons. A new oscillation camera with high mechanical precision and fast rotation speed is described.

**Keywords: macromolecular crystallography; oscillation camera.**

## 1. Introduction

The macromolecular oscillation cameras that have been in service over the last 12 years at CHESS were constructed as modular units based on an optical rail (Klinger X-95). These cameras consist of a precision rotation stage (Huber 410) driven by a 200 steps revolution$^{-1}$ stepping motor (Slo-Syn No. M061-FC02E) which has a shaft-mounted rotary encoder (Hewlett Packard No. HEDS-6310). A 10:1 gear reducer (Huber No. 2013) is used to give a stage angular resolution of $5 \times 10^{-4}$ °. The stepping motors on these cameras are controlled by a driver (Advanced Control Systems MDU-8) which allows angular speeds of 0.2–2° s$^{-1}$.

In recent years two developments have placed new challenges on the cameras. Firstly, it has been routinely observed that 100 μm crystals are well behaved with regards to cooling to liquid nitrogen temperatures. Such crystals are frequently found to give the highest-quality diffraction data. Small samples place stringent requirements on the camera mechanism. Secondly, X-ray intensities have significantly increased. Present intensities frequently require crystal-rotation speeds much greater than 2° s$^{-1}$ to avoid saturating the detector. The need for high rotation speed is further strengthened by the popularity of MAD phasing in which the crystal is frequently rotated through 180° to collect Friedel mates. With existing cameras, this rotation may require 90 s.

## 2. Description of the camera

### 2.1. *General*

The camera components are directly attached to a precision-ground table top to achieve maximum mechanical stability and precision alignment. Two rails (THK No. SR-20W), each with four-row recirculating bearing raceways, serve to align and position the various components precisely. These include the housing which holds the X-ray shutter, the X-ray collimator and the optical system for viewing the sample, the goniostat for positioning the sample, the beamstop, the detector for collecting the diffraction patterns, the nozzle which directs cold gaseous nitrogen at the sample, and the fluorescence detector for MAD measurements. The camera is shown in Fig. 1. The table top is attached to a sub-frame with stepping-motor-driven stages providing six degrees of positional freedom. The sub-frame allows translational positioning of the table as fine as 1.2 μm and overall rotations as small as 0.002 mrad.

Control software has been developed which allows the table top, along with the entire oscillation camera, to be rotated about any point in space. Typically, this point is defined as the input of the X-ray collimator. While the system is being aligned to the incident beam, the alignment of the individual camera components is preserved.

### 2.2. *Shutter housing*

The main housing serves to hold the collimator, the X-ray shutter (along with the necessary electronics associated with it), an ion chamber to monitor the flux before the collimator and the optical system for viewing the crystal sample. The housing shields stray radiation from the detector. As the X-ray beam enters the 13 mm-diameter entrance of the housing, it first passes through a high-voltage ion chamber to monitor the flux of the full beam. Next, the beam encounters the X-ray shutter. As exposures are typically 20 s or less in duration and each exposure may consist of several oscillations of the crystal, this shutter must be very robust as well as accurate in its timing. The system which has proven to be best suited for this application is a rotary solenoid. Attached to a 24 V rotary solenoid (Trytech Electronics Dist#H-2616–0339628) is a plate with a tungsten block which serves to shield the X-ray when the solenoid is in the non-energized position. The other side of the solenoid has an aluminium plate supporting a blade which triggers a signal from a photodiode switch to monitor the status and timing of the shutter. A schematic diagram of the X-ray shutter unit is shown in Fig. 2. The opening speed of the shutter is 20 ms.

A spring-loaded mechanism locks the 7.6 cm-long X-ray collimator (Huber) into position. Quick and precise interchange of collimators is allowed. To reduce X-ray scatter, an adjustable tube made of tungsten–copper alloy slides over the input end of the collimator. The typical collimator size presently used with this camera is 100 μm. Efforts are underway to routinely operate with 20 μm collimators made from untapered glass capillary tubing. In the future, even smaller collimators (Thiel *et al.*, 1992) or tapered concentrators (Bilderback & Thiel, 1995) may be incorporated.

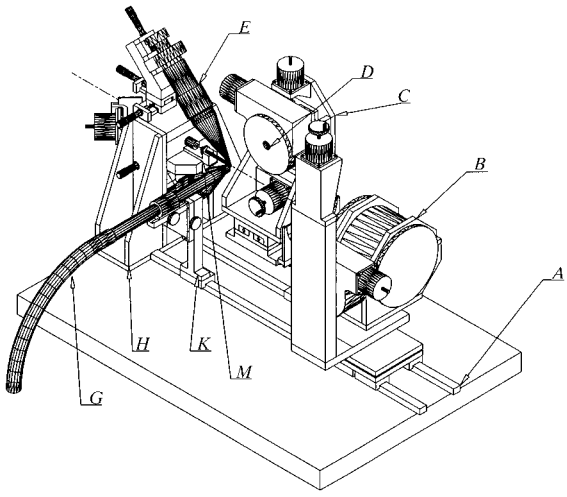


**Figure 1**
Components of the oscillation camera: *A*, precision rails; *B*, CCD detector; *C*, goniostat support; *D,* rotation stage; *E*, crystal-viewing optics; *G*, cold-stream nozzle; *H*, shutter housing; *K*, nozzle support; *M*, collimator holder.

### 2.3. *Shutter control electronics*

A beamline-control computer controls all motors and counters at the beamline. The control software used is *SPEC* (Certified Scientific Software) running on a UNIX workstation. With this system, reliable real-time experimental control is impossible to achieve without external hardware adaptations. A robust system capable of controlling the precision shutter/motor interactions required for high-quality crystallographic data has been constructed from readily available hardware components and simple electronics. The control electronics will be described elsewhere (LaIuppa, 1997).

### 2.4. *Goniostat*

The sample is mounted onto a goniometer head which allows centring of the sample within the X-ray beam. Limited orientation of the crystal may be achieved by adjusting the goniometer head. This component attaches to a larger system of rotation stages known as the goniostat. Standard diffractometers are made up of one, two or four rotation circles to allow various degrees of control over the experiment.

At CHESS, all macromolecular crystallographic data are collected using a single-axis system due to its effectiveness, simplicity and low cost. By convention, this axis is referred to as the $\varphi$ axis. The camera described here provides rotation about the $\varphi$ axis. With modifications, additional rotation circles could be added, giving a $\kappa$-axis system. The benefit of such a system would be primarily for anomalous scattering applications including MAD phasing.

A rotation stage (Newport No. RTM-80PP) having high speed capability ($40^\circ\ s^{-1}$), good repeatability ($0.002^\circ$) and compact size was chosen.† The precision of this stage assures that samples as small as 2 µm will remain centered in the beam as the sample is rotated about $\varphi$. To accommodate the need for fast crystal rotation while maintaining very fine angular resolution, the stage is driven by a system capable of microstepping. A VME controller (Oregon MicroSystems VME58) capable of operating at 1 MHz sends pulses to a stepping motor driver (Centent CN0165) which operates a 200 steps revolution$^{-1}$ motor (Step-Syn 103 H548-0440). The motor is directly coupled to the worm-drive of the rotation stage where one motor revolution corresponds to a $2^\circ$ stage rotation.

The camera has provision to switch easily to the Huber 410 stage. Particular cases where this change is of value will be described elsewhere (Thiel, Appleby *et al.*, 1997).

The controller can operate in the conventional mode where each pulse corresponds to a motor step, or it can be microstepped so that up to 250 controller pulses drive the motor one step, giving 50000 microsteps per motor shaft revolution. Microstepping reduces the need for mechanical gear reduction to attain precise angular resolution; hence, it allows both fine resolution as well as rapid angular speed. In the mode where 250 controller microsteps equals one motor step, angular speeds from $0.4^\circ\ s^{-1}$ to $30^\circ\ s^{-1}$ are achieved with a step resolution of $4 \times 10^{-5}$ deg.

### 2.5. *Sample manipulation*

Several features have been incorporated into the camera design to accommodate tasks involving sample manipulation such as crystal mounting, viewing and freezing. The goniostat is supported by cross roller bearing translations allowing 2.5 cm of travel vertically and 7.6 cm of travel horizontally. Once aligned, the camera allows the use of various goniometer heads including low-profile translation-only heads, heads with standard arcs and heads with extended arcs as well as different crystal-mounting components. The horizontal translation is collinear with the $\varphi$-axis to within 0.2 mrad. Opposite the goniostat is a mount which holds the cryo-stream nozzle (Molecular Structure Corporation No. THM231) collinear with the sample for optimized laminar flow and hence minimal ice formation. This mount also provides translation along the $\varphi$ axis to assist in the manipulation of crystals suspended in propane which has been solidifed by cooling to 77 K. An additional function of this mount is to support a fluorescence detector (Amptek XR-100T) for MAD phasing experiments.

For crystal centring without sample rotation two cameras are mounted perpendicular to one another, with the collimator bisecting the $90^\circ$ angle. One camera (Sony SSC-C370) has color contrast and magnification from $18\times$ to $100\times$. This camera alone is often used for crystal centring where the goniostat is rapidly rotated by the experimenter using a hand-held control switch. Variations in position seen with the previous cameras upon unlocking the mechanical drive are thereby eliminated.

### 2.6. *Detectors*

Attached to the primary rails is a stage with a kinematic mount which can support any of several detectors. The crystal-to-detector distance is calibrated by collecting diffraction data. Once calibrated, the reproducibility of the mount allows the interchange of detectors without losing calibration. An encoder on the stage sends a signal to the control computer in order to have the exact positioning of the detector known at all times.

The detectors used with the camera include image plates (Fuji HRIIIn) as well as CCD detectors. For image plates, a six-plate carousel made of a Huber 410 stage fastens to the detector stage. More frequently, the efficient CCD detectors are desired. The carousel is removed from the mount and any of three CCD detectors may be readily inserted into place. The three CCD systems are a 1k × 1k system (Tate *et al.*, 1995), a 2k × 2k system (Thiel *et al.*, 1996) and a newer 1k × 1k system (Area Detector Systems Corporation Quantum-1). The detectors may be translated horizontally or vertically as well as tilted about the spindle axis in a $2\theta$ fashion. In the near future, a mosaic CCD (Area Detector Systems Corporation Quantum-4) will be incorporated. The integrity of the rail system assures that the additional weight of the mosaic detector can be accommodated.

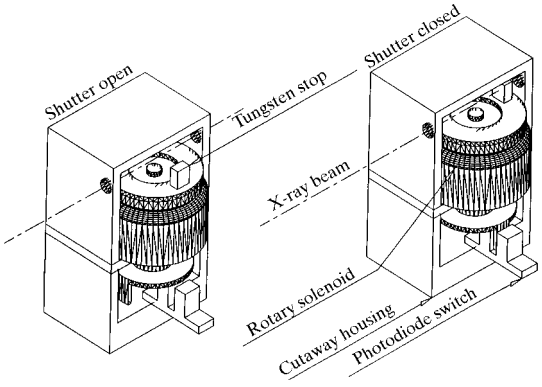


**Figure 2**
X-ray shutter system.

† *Note added in proof*: The Newport rotation stage exhibited mechanical wear after several months and has been replaced by a Huber 410 stage.

### 2.7. *Beamstops and protective shutter*

Two beamstop designs are used. The first is a small lead disk, typically 1 mm in diameter, mounted onto a vertical strip of kapton tape. Minimal diffraction data are blocked, but the system is delicate. A more robust design is a low-profile horizontal bar. The loss of data in the horizontal due to shadowing from the bar is insignificant due to high Lorentz correction along this region.

Because the beamstop must be versatile to enable the experimenter full access to the region around the sample, it cannot serve as the primary safety mechanism preventing direct exposures of the detector when damage is possible, as is the case with CCD detectors. Instead, a robust protective system is used. The system will be described elsewhere (Thiel, Blank *et al.*, 1997).

## 3. Conclusions

The camera was installed on the F2 beamline at CHESS in November 1996. Well over 100 data sets have been collected; the quality of the data is very high. The integrity and speed of the camera have been proven by the fact that the number of MAD structures solved during the initial six-month period equals the total number of MAD structures solved at CHESS prior to this run. A complete evaluation of the performance of the camera will be presented elsewhere (Thiel, Appleby *et al.*, 1997).

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