

Experiences with CCD detectors on a home X-ray source

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Charged-coupled device (CCD) detectors have been widely accepted as detectors for collecting X-ray diffraction images. The CCD detector offers a sensitive detection system well suited for diffraction analysis and, compared with other detectors on the market, a relatively rapid system for read-out of the collected image. The two predominant markets for the CCD detector have been those in which relatively short exposure times are used, *i.e.* small-molecule X-ray diffraction and large-molecule crystallography at high-intensity synchrotron sources. CCD detectors have not been commonly used on rotating-anode X-ray sources for large-molecule crystallography. Comparison of the performance of the CCD detectors with commercially available image-plate detectors shows that the CCD detectors function in a similar fashion to image-plate-based detectors.

1. Introduction

CCD detectors have been used as area detectors in crystallographic applications since their development in the late 1980s. The driving force for the development of these detectors was the desire to have a detector which would allow rapid read-out of the diffraction image combined with large reciprocal-space coverage. The theory of operation of a CCD detector for use in crystallography is beyond the scope of this paper; however, excellent reviews have been published (Westbrook & Naday, 1997).

The CCD detector has become the detector of choice for synchrotron applications, and a number of commercial equipment manufacturers offer CCD detectors in a wide array of configurations which have suitable software for collecting diffraction data. In an effort to increase coverage of reciprocal space, detector makers have designed cameras using either arrays consisting of multiple copies of CCD chips or employing large single-module CCD chips.

2. Detector comparison

In this section, CCD detectors are compared with other commercially available detectors, with regard to the performance of these detectors on rotating-anode sources. The areas in which these detectors are compared are speed, reciprocal-lattice coverage and data quality.

2.1. Speed

Fig. 1 shows a graph of the calculated speeds of a theoretical CCD detector and two image-plate detectors commonly employed in home-laboratory sources, the MAR 345 and the R-AXIS IV image-plate detectors, as a percentage of the total

time spent during data collection. The plot shows that CCD detectors are significantly faster data-collection instruments than imaging plates. The most significant differences in speed occur at exposure times less than 5 min or so, and it is this speed advantage which has made CCD-based detectors the best suited for use at synchrotron sources, where exposure times are commonly found on the time scale of 10 s or less. In the home laboratory, given the typical flux obtainable from rotating-anode sources, the exposure times are usually in the 1–10 min range and the percentage advantage in speed for the CCD is less significant. However, data-collection regimes in which small oscillation angles are used for each exposure will benefit from the CCD detector's rapid read-out.

2.2. Reciprocal-lattice coverage

Fig. 2 illustrates the coverage of a 133 mm CCD detector and a 300 mm image-plate detector. The resolution limits were calculated assuming that the detector is at a 2θ angle equal to zero, using Cu $K\alpha$ (1.54 Å wavelength) radiation. From this plot, it is clear that current image-plate technology can provide much better coverage of reciprocal space than CCD detectors. However, for many projects in macromolecular crystallography, the diffraction pattern is limited to much less than atomic resolution. In drug-design applications, it is generally accepted that data to 2.5 Å resolution is adequate for most protein–ligand structure determinations (Verlindé & Hol, 1994). CCD detectors in general have slightly better spatial resolution than image-plate detectors and, if properly matched with small cross section X-ray beams, can give reciprocal-space coverage comparable to an image-plate detector.

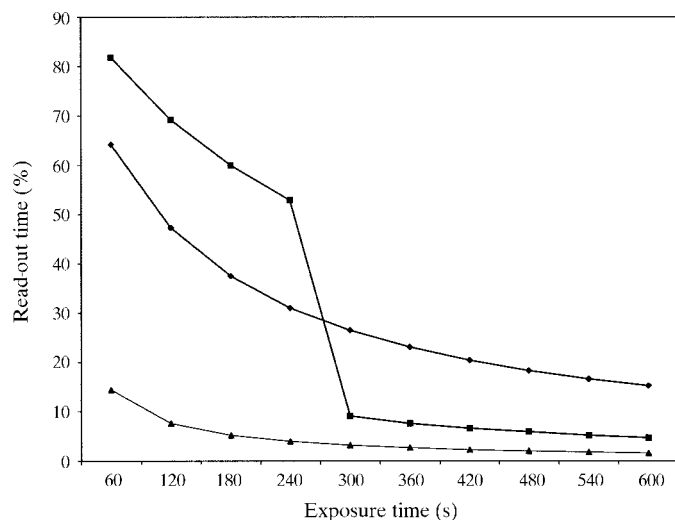


Figure 1 Comparison of percent time spent in read-out *versus* exposure time. Three detectors are shown here, the MAR 345 image-plate detector (diamonds; 108 s dead time), the R-AXIS IV image-plate detector (squares; 235 s dead time) and a theoretical CCD (triangles; 10 s dead time). The discontinuity of the R-AXIS IV plot is a consequence of the detector utilizing two imaging plates, so that when the exposure time is greater than the cycle time, the dead time equals simply the positioning and erase time.

2.3. Data quality

In order to investigate the relative data quality obtainable from a CCD detector with a home-laboratory source, a data-collection experiment was performed in which all controllable experimental conditions were reproduced identically on both a CCD-detector system and an image-plate system. The results of these two data collections are summarized in Figs. 3 and 4. The diffraction data was collected from the same hen egg-white lysozyme crystal, mounted at room temperature in an X-ray capillary, using identical Osmic multilayer optics on opposing ports of a Rigaku RU-200 X-ray generator. The

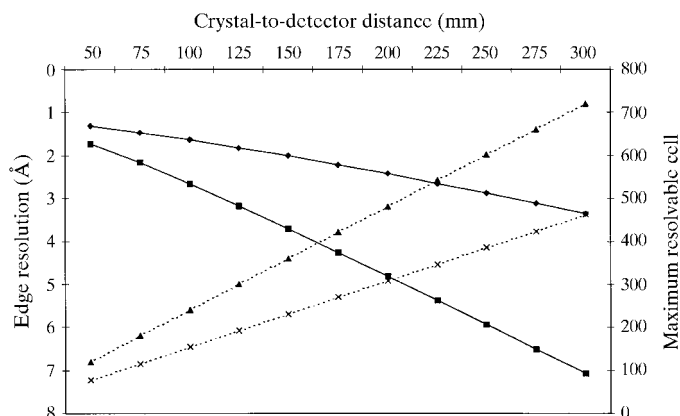


Figure 2 Edge resolution limits and corresponding maximum unit-cell dimensions resolvable for both a 133 mm CCD detector (0.064 mm pixel size) (edge shown as squares, cell dimensions as triangles) and a 300 mm diameter image-plate detector (0.1 mm pixel size) (edge shown as diamonds, cell dimensions as crosses) using Cu $K\alpha$ (1.54 Å) radiation and calculated at crystal-to-film distances commonly used in macromolecular crystallography. The largest unit-cell repeat distance resolvable was calculated for both detectors assuming a minimum of 10 pixels would be required between diffraction maxima to resolve the spots.

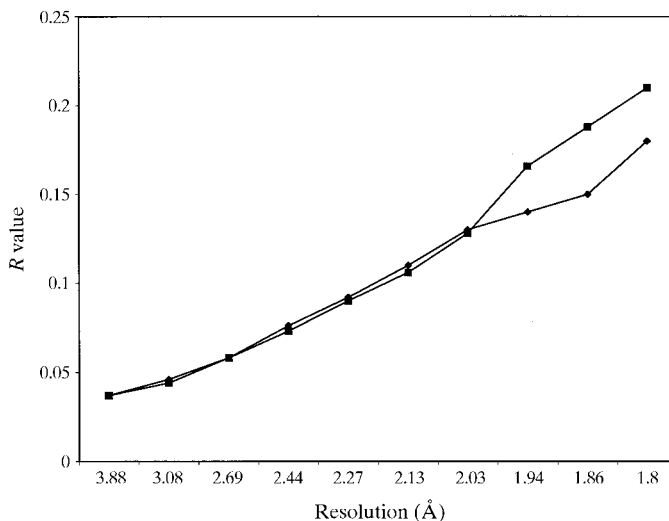


Figure 3 R-value plot for lysozyme data collected on a CCD detector (diamonds) and an image-plate detector (squares). The R value (linear R value on intensity) plotted *versus* resolution curves are similar for the two detector types.

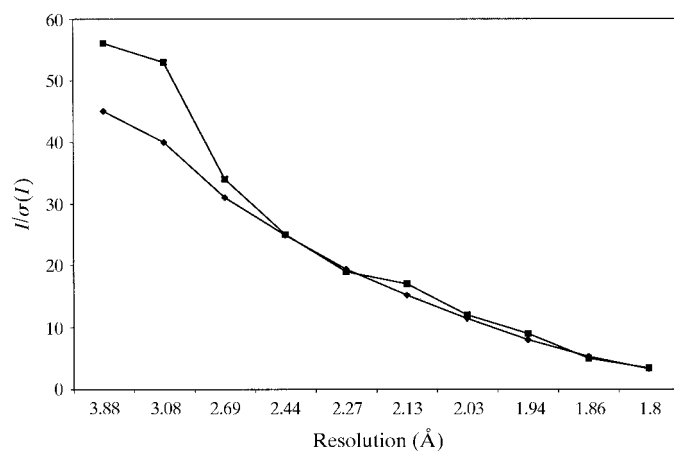


Figure 4

$I/\sigma(I)$ plots for data collected on an IP (diamonds) and a CCD detector (squares). The curves show good agreement throughout the range for the estimated signal from these two detectors.

detectors used were a 300 mm diameter MAR Research image plate and a 133 mm diameter MAR USA CCD detector. The detector systems were mounted on identical goniostat arrangements, supplied by MAR Research, and fitted with alignment slits and ionization gauges to estimate the intensity of the incident X-ray beam. The distance for each detector was selected so the edge of the each crystal detector was approximately 1.8 Å resolution, *i.e.* 80 mm crystal-to-film distance for the CCD detector and 110 mm crystal-to-film distance for the image-plate detector. The data-collection parameters were: oscillation angle, 0.5°; exposure time, 20 s; total data collected, 45°. The IP data were recorded first and the crystal was then transferred to the CCD detector, where the experiment was repeated over the same angular range with the same data-collection parameters as the IP data collection. The *HKL* suite (*XDISPLAYF*, *DENZO* and *SCALEPACK*) of data-reduction programs (Otwinowski & Minor, 1997) were used to reduce the resultant data from both detectors.

As seen in Figs. 3 and 4, the data collected from these two detectors are essentially identical. The merging R values for the data from the two detectors are essentially identical, not only in their overall values, but in their values in each resolution bin. This trend is also observed for the $I/\sigma(I)$ plots. In addition, Fig. 5 shows the R value, calculated on I , comparing the two data sets. This curve shows good agreement for the two data sets, with an overall value of 2.1% between the two

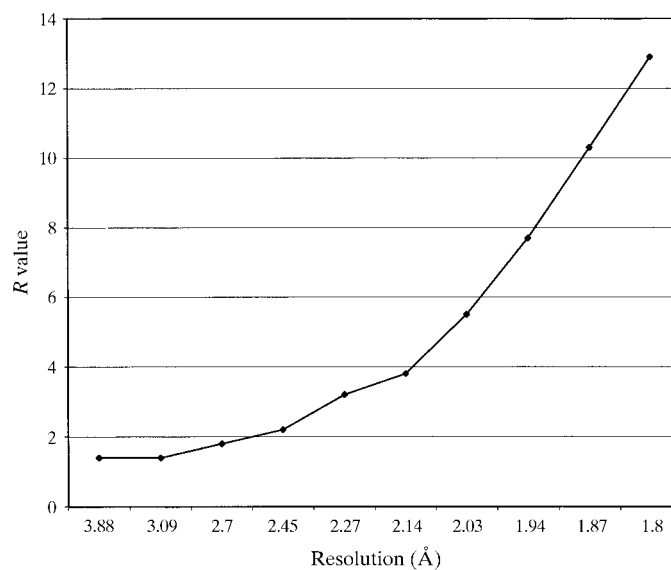


Figure 5

Agreement statistics for data collected on an IP and a CCD detector. The reduced data sets were compared using the *SCALEPACK* program (Otwinowski & Minor, 1997) and the R value plotted is the linear R value on intensity.

sets. The major difference observed for these two data-collection experiments was that less than an hour was needed to collect the CCD set compared with over 4 h to collect the image-plate data set.

3. Conclusions

CCD detectors have gained wide acceptance as instruments for the collection of diffraction data from both macromolecular and small-molecule crystals. The detectors are suited for both synchrotron and home-laboratory X-ray sources, and experience in our laboratory has shown that CCD detectors can be used to collect useful macromolecular data on home sources.

References

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