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7. X-RAY DETECTORS

7.1. Comparison of X-ray detectors

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7.1.1. Commonly used detectors: general considerations

This chapter summarizes detector characteristics and provides practical advice on the selection of crystallographic detectors. Important types of detectors for crystallographic applications are summarized in Section 7.1.2 and listed in Table 7.1.1.1. To be detected by any device, an X-ray must be absorbed within a detective medium through electrodynamic interactions with the atoms in the detecting layer. These interactions usually result in an energetic electron being liberated which, by secondary and tertiary interactions, produces the signal that will be measured, *e.g.* luminescence in phosphors, electron–hole pairs in semiconductors or ionized atoms in gaseous ionization detectors. As we shall see, there are many schemes for recording these signals. The various detector designs, as well as the fundamental detection processes, have particular advantages and weaknesses. In practice, detector suitability is constrained by the experimental situation (*e.g.* home laboratory X-ray generator *versus* synchrotron-radiation source; fine-slicing *versus* large-angle oscillations), by the sample (*e.g.* whether radiation damages it readily) and by availability. An assessment of detector suitability in a given situation requires an understanding of how detectors are evaluated and characterized. Some of the more important criteria are discussed below.

The *detective quantum efficiency* (DQE) is an overall measure of the efficiency and noise performance of a detector (Gruner *et al.*, 1978). The DQE is defined as

$$\mathrm{DQE} = (S_o/N_o)^2/(S_i/N_i)^2, \qquad (7.1.1.1)$$

where S is the signal, N is the noise, and the subscripts o and i refer to the output and input of the detector, respectively. The DQE measures the degradation owing to detection in the signal-to-noise ratio. For a signal source that obeys Poisson statistics, the inherent noise is equal to the square root of the number of incident photons, so that the incident signal-to-noise ratio is just $S_i/N_i = (S_i)^{1/2}$. The ideal detector introduces no additional noise in the detection process, thereby preserving the incident signal-to-noise ratio, *i.e.* $\mathrm{DQE} = 1$. Real detectors always have $\mathrm{DQE} < 1$ because some noise is always added in the detection process. The DQE automatically accounts for the fact that the input and output signals may be of a different nature (*e.g.* X-rays in, stored electrons out), since it is a ratio of dimensionless numbers.

A single number does not characterize the DQE of a system. Rather, the DQE is a function of the integrated dose, the X-ray spot size, the length of exposure, the rate of signal accumulation, the X-ray energy *etc.* Noise in the detector system will limit the DQE at low dose, while the inability to remove all systematic nonuniformities will limit the high-dose behaviour.

The *accuracy*, ρ, measures the output noise relative to the signal, *i.e.* $\rho = N_o/S_o$. For a Poisson X-ray source, it follows that the accuracy and the DQE are related by

$$\rho = (N_i\mathrm{DQE})^{-1/2}. \qquad (7.1.1.2)$$

This allows the determination of the number of X-rays needed to measure a signal to a given accuracy with a detector of a given DQE. The accuracy for an ideal detector is $1/(S_i)^{1/2}$, *e.g.* 100 X-rays are required to measure to 10% accuracy, and 10^4 X-rays are needed for a 1% accuracy. Nonideal detectors $(\mathrm{DQE} < 1)$ always require more X-rays than the ideal to measure to a given accuracy.

Spatial resolution refers to the ability of a detector to measure adjacent signals independently. The spatial resolution is characterized by the *point spread function* (PSF), which, for most detectors, is simply the spread of intensity in the output image as a result of an incident point signal. An alternative measure of resolution is the *line*

Table 7.1.1.1. *X-ray detectors for crystallography*

(*a*) Commercially available detectors

Technology	Primary X-ray converter	Format
Film	AgBr	Area
Storage phosphor	BaFBr	Area
Scintillating crystal	NaI, CsI	Point
Gas discharge	Xe	Point, linear, area
Television	Phosphor	Area
CCD	Phosphor	Area
Silicon diode	Si	Linear, area
Avalanche diode	Si	Point, area

(*b*) Detectors under development

Technology	Primary X-ray converter	Format
Pixel array	Si, GaAs, CdZnTe	Area
Amorphous silicon flat panel + phosphor	CsI, Gd_2O_2S	Area
Amorphous silicon flat panel + photoconductor	PbI_2, CdZnTe, TlBr, HgI_2	Area

spread function (Fujita *et al.*, 1992). Although a detector might have a narrow PSF at 50% of the peak level, poor performance of the PSF at the 1% level and below can severely hamper the ability to measure closely spaced spots. It is important to realize that the PSF is a two-dimensional function, which is often illustrated by a graph of the PSF cross section; therefore, the integrated intensity at a radius R pixels from the centre of the PSF is the value of the PSF cross section times the number of pixels at that radius. Often the wings of the PSF decay slowly, so that considerable integrated signal is in the image far from the spot centre. In this case, a bright spot can easily overwhelm a nearby weak spot. Another consequence is that bright spots appear considerably larger than dim ones, thereby complicating analysis.

The *stopping power* is the fraction of the incident X-rays that are stopped in the active detector recording medium. In low-noise detectors, the DQE is proportional to the stopping power. A detector with low stopping power may be suitable for experiments in which there is a strong X-ray signal from a specimen that is not readily damaged by radiation. On the other hand, even a noiseless detector with a low stopping power will have a low DQE, because most of the incident X-rays are not recorded.

Unfortunately, many definitions of *dynamic range* are used for detectors. For an integrating detector, the dynamic range per pixel is taken to be the ratio of the saturation signal per pixel to the zero-dose noise per pixel for a single frame readout. For photon counters, the dynamic range per pixel refers to the largest signal-to-noise ratio, *i.e.* the number of true counts per pixel that are accumulated on average before a false count is registered. In practice, the dynamic range is frequently limited by the readout apparatus or the reproducibility of the detector medium. For example, the large dynamic range of storage phosphors is almost always limited by the capabilities of the reading apparatus, which constrains the saturation signal and limits the zero-dose noise by the inability to erase the phosphor completely. The number of bits in the output word does not indicate the dynamic range, since the number of stored bits can only constrain the dynamic range, but, obviously, cannot increase it.

The dynamic range is sometimes given with respect to an integrated signal that spans more than one pixel. For a signal S per pixel which spans M pixels, the integrated signal is MS, and, assuming the noise adds in quadrature, the noise is $N(M)^{1/2}$, yielding a factor of $(M)^{1/2}$ larger dynamic range. For most detectors, the noise in nearby pixels does not add in quadrature, so this is an upper limit.

The characteristics of a detector may be severely compromised by practical considerations of *nonlinearity*, *reproducibility* and *calibration.* For example, the optical density of X-ray film varies nonlinearly with the incident dose. Although it is possible to calibrate the optical density *versus* dose response, in practice it is difficult to reproduce exactly the film-developing conditions required to utilize the highly nonlinear portions of the response. A detector is no better than its practical calibration. This is especially true for area detectors in which the sensitivity varies across the face of the detector. The proper calibration of an area detector is replete with subtleties and constrained by the long-term stability of the calibration. Faulty calibrations are responsible for much of the difference between the possible and actual performance of detectors (Barna *et al.*, 1999).

The response of a detector may be nonlinear with respect to position, dose, intensity and X-ray energy. Nonuniformity of response across the active area is compensated by the *flat-field* correction. Frequently, nonuniformity of response varies with the angle of incidence of the X-ray beam to the detector surface, which is a significant consideration when flat detectors are used to collect wide-angle data. Although this may be compensated by an energy-dependent *obliquity* correction, few detector vendors provide this calibration. An X-ray image may also be spatially distorted; this *geometric distortion* can be calibrated if it is stable.

Other important detector considerations include the *format* of the detector (*e.g.* the number of pixels across the height and width of the detector). The format and the PSF together determine the number of Bragg orders that can be resolved across the active area of the detector. *Robustness* of the detector is also important: as examples, gas-filled area detectors may be sensitive to vibration of the high-voltage wires; detectors containing image intensifiers are sensitive to magnetic fields; or the detector may simply be easily damaged or lose its calibration during routine handling. Some detectors are readily damaged by too large an X-ray signal. *Count-rate* considerations severely limit the use of many photon counters, especially at synchrotron-radiation sources. Detector *speed*, both during exposure and during read out, can be important. Some detector designs are highly *flexible*, permitting special readout modes, such as a selected region of interest for use during alignment, or operation as a streak camera.

Ease of use is especially important. A detector may simply be hard to use because, for example, it is exceptionally delicate, requires frequent fills of liquid nitrogen, or is physically awkward in size. A final, often compelling, consideration is whether a detector is *well integrated into an application* with the appropriate analysis software and whether the control software is well interfaced to the other X-ray hardware.

7.1.2. Evaluating and comparing detectors

The DQE comprehensively characterizes the ultimate quantitative capabilities of an X-ray detector. The DQE may be determined from an analysis of the reproducibility of recorded X-ray test images of known statistics *via* equation (7.1.1.1): given M incident X-rays per exposure, the expected incident signal-to-noise is $(M)^{1/2}$. The DQE is determined by measuring the variance in the recorded signal in repeated measurements of the test image. Repetition of this process for different values of M maps out the DQE curve. Since the DQE is dependent on the structure of the image, the integration area, the X-ray background and the long-term detector calibration, it is essential that the test images realistically simulate these features as expected in experiments. Thus, if the detector is to be used to obtain images of diffraction spots, the test images should consist of comparably sized spots superimposed on a suitable background.

A comprehensive DQE determination is nontrivial and requires specialized tools, such as test masks, uniform X-ray sources *etc.* Unfortunately, published DQE curves are frequently incorrect and misleading. Users can, however, set up and perform a simple DQE assessment, detailed below, which gives a great deal of information about the sensitivity and usefulness of a given area detector. Other sources of stable X-ray spots (of appropriate size and intensity) can also be used in similar tests.

The materials needed are sheet lead and aluminium, a sewing needle, a stable collimated X-ray source, X-ray capillaries filled with saturated salt solutions, an X-ray shutter with timing capability and a scintillator/phototube X-ray counting arrangement. Arrange a fluorescent X-ray source to provide a diffuse X-ray signal. An X-ray capillary filled with a saturated solution of iron chloride makes a suitable source for a copper anode machine. Next, make an X-ray-opaque metal mask by punching a clean pinhole with a sewing needle in a lead sheet. The size of the hole should be representative of an X-ray spot, say 0.3 mm in diameter. The mask should be firmly and reproducibly secured a few cm from the fluorescent source at a wide angle to the incident beam. Using a scintillator/phototube combination, measure the number of X-rays per second emerging through the hole at a given X-ray source loading. A sufficient number of X-rays per measurement (say 10^5) is necessary

to obtain accurate statistics (0.3%). This measurement should be repeated to verify the stability of the source.

This spot can now be recorded by the detector in question, using different integration times to vary the dose. 20 measurements at each integration time should give a reliable measure of the standard deviation in the signal. It is vital to move the position of the spot on the detector face for each exposure, taking care to move only the detector without disturbing the remainder of the experimental setup. Only by moving the detector is the fidelity of the calibrations tested. One subtlety is that the sensitivity of many detectors varies with the angle of incidence of the X-rays, so that it will be necessary to vary both the position and angle of the detector between exposures.

By using a wide range of integration times, both the sensitivity of the detector at low doses and the ultimately achievable measurement accuracy can be examined. These data may also highlight specific problems a detector might have, such as nonlinearity.

The DQE can be measured for a spot in the presence of a background if the lead pinhole mask is now replaced with a pinhole in a semitransparent aluminium foil. Choose the foil thickness to yield an appropriate background level, say 20% of the pinhole intensity. The uncertainty in the measurement of the spot intensity now results from the total counts in the integration area in addition to the uncertainty in determining the background. A wide PSF is especially harmful in this case, since many more pixels must be integrated to encompass the spot.

These evaluation procedures test only limited aspects of the detector, but in doing so, much is learned not only about the detector, but also about the degree to which the vendor is willing to work with the user, which is clearly of interest. The ultimate test for a crystallographer is whether a detector delivers good data in a well understood experimental protocol. Usually, values of $R_{\rm sym}$, the agreement of integrated intensities from symmetry-related reflections, are evaluated as a function of resolution. Low values of $R_{\rm sym}$ suggest good quality data. A much more stringent test can be made by comparing anomalous difference Patterson maps based on the Fe atom in myoglobin (Krause & Phillips, 1992). The limitation in these crystallography-based evaluations is that they tend to rely on robust, strongly diffracting crystals, which allow accumulation of good X-ray statistics even with insensitive detectors. Weakly diffracting and radiation-sensitive crystals are less forgiving.

7.1.3. Characteristics of different detector approaches

7.1.3.1. *Point versus linear versus area detection*

A point detector may be based on a scintillating crystal or a gas-filled counter, with the sensitive area defined by slits or a pinhole mask. The spatial resolution of such a detector can be made arbitrarily fine at the expense of data collection rate. Point detectors can have very high accuracy if the background is removed by energy discrimination. They find application in powder diffractometry and small-molecule crystallography, in which the reflections are widely dispersed, thereby simplifying measurement of individual reflections. Clearly, specimen and source stability are important for such work.

Throughput can be greatly increased by area detection, which is often required for macromolecular crystallography or investigations of unstable specimens. Typical area detectors, such as film, storage phosphors and charge-coupled devices (CCDs), are described below.

7.1.3.2. *Counting and integrating detectors*

Detectors can be broadly divided into photon counters and photon integrators. Photon counters have the advantage that some designs permit energy discrimination, allowing them to reject inelastically scattered radiation, thereby improving the signal-to-noise ratio. However, photon-counting detectors always have a count-rate limitation, above which they begin to miss events, or even become unresponsive (the time during which a detector misses events is known as dead time). Prototype systems have demonstrated linear count rates greater than 10^6 photon s^{-1}. Fabrication difficulties have limited the commercial availability of photon-counting detectors with large areas, high spatial resolution and high count rates. The count rate is a particular concern at modern synchrotron sources, which are capable of generating diffraction that delivers two or more photons to a pixel during one bunch time, an instantaneous count rate greater that 10^{10} photons per second per pixel. Integrating detectors are more typically used in situations where very high event rates are expected.

In contrast, integrating detectors have no inherent count-rate limitation, though at very high fluxes several sources of nonlinearity can theoretically become important, such as nonlinearity in the phosphor used to convert the X-ray image to a visible image. Integrating detectors, however, do not discriminate energy, and they have noise that increases with integration time. Nonetheless, film, image-plate and CCD integrating detectors are currently commercially available and in widespread use.

7.1.3.2.1. *Photon-counting detectors*

Commonly used photon counters include *scintillator/photomultiplier combinations*, *gas-filled counters* and *reverse-biased semiconductor detectors*.

Scintillator/photomultipliers usually consist of a relatively thick crystal of a scintillator coupled to a high-gain photomultiplier tube. These detectors are generally designed to serve as point photon counters with moderate energy resolution. In order to perform this function, several constraints must be met:

(1) The scintillator crystal must be thick enough to have almost unity stopping power.

(2) It is necessary to collect as many of the converted visible photons as possible, so an optically clean scintillator crystal is used in a reflective housing to direct as many photons as possible toward the phototube.

(3) The scintillator must emit its light quickly, so as to minimize dead time, and be efficient, so as to emit much light. NaI:Tl, CsI:Na and CsI:Tl meet these constraints. NaI is more commonly used, but CsI may be preferred at higher X-ray energies because of its higher stopping power. Both materials are hygroscopic and are usually encased in hermetically sealed capsules with beryllium windows.

(4) The phototube is usually operated in its linear region for energy discrimination.

Scintillator/phototube combinations are relatively trouble-free and often have near-unity DQE. Their main limitations are count rates well below 10^6 photon s^{-1} and the lack of spatial resolution. Even so, such detectors are still preferred in many applications where the data are effectively zero- or one-dimensional.

Reverse-biased semiconductor detectors are designed to have a thick depletion zone in which charge can be efficiently collected and conveyed to an amplifier. X-rays that stop in the depletion zone produce electron–hole pairs; these are separated by the depletion zone field and the electrons are swept to the input of a low-noise amplifier. Single-photon counting can be readily achieved, even for low-energy X-rays, especially if the detector is cooled to minimize thermally generated charge. These detectors are typically fabricated as silicon diodes, but germanium and gallium arsenide are also used (Hall, 1995). Until recently, these devices were generally configured as point detectors or strip detectors consisting of a linear array of narrow sensitive regions, forming a one-dimensional detector (Ludewigt *et al.*, 1994). Two-dimensional arrays of square pixels are being developed, *e.g.* see the description of pixel array

detectors below. In another device, the silicon drift detector, potentials are arranged in the silicon to funnel signals from a large area to a low-noise collection point (Rehak *et al.*, 1986). Such devices are being developed for both linear and area applications.

By increasing the electric field strength in an appropriately designed p-n junction in silicon, avalanche multiplication of the X-ray-induced electrons can be obtained as they move toward the anode where they are collected. This gives rise to a very high linear signal gain, with high speed and low noise. Arrays of such *avalanche photodiodes* as large as 8×8 elements, each 1×1 mm square, have been fabricated (Gramsch *et al.*, 1994; Farrell *et al.*, 1994).

Gas discharge (wire) counters make use of the ionization produced when an X-ray is stopped in the high-atomic-number gas, usually xenon, that fills the detector. A strong electric field between a fine anode wire and a cathode plane accelerates the products of the primary ionization to produce an ionizing multiplication (either a proportional or an avalanche discharge, depending on the field strength) that is detected as a charge pulse on one or both of the electrodes. The discharge is quenched by the presence of a few per cent of a second gas, *e.g.* methane or carbon dioxide. Gas discharge detectors have been configured in zero-, one- and two-dimensional versions and continue to be widely used in some applications. The venerable Geiger counter is in this class and is used for radiation monitoring and beam alignment in home laboratories. Properly designed gas discharge counters have very low noise, but the quantum efficiency depends critically on design, gas and X-ray energy.

Linear wire detectors have been used to record small-angle X-ray scattering. The localization of the X-ray event along the length of the detector is often performed by measuring the difference in arrival time of the charge pulses at the two ends of one of the electrodes (Barbosa *et al.*, 1989). The pulses are stretched to permit this measurement. One design uses a resistive anode wire to perform this function, whereas others configure the cathode plane as a delay line. Various two-dimensional arrangements of crossed planes of wires, broadly classified as multiwire proportional counters (MWPCs), have been widely used in crystallography, and some types have been commercially successful (Hamlin *et al.*, 1981; Blum *et al.*, 1987).

The design of MWPC area detectors has had difficulty keeping up with improvements in X-ray sources, particularly the high fluxes available at storage rings, and the shift toward use of higher-energy X-rays. The electric discharge at the heart of the technology has an inherent dead time associated with it. Added to this inherent dead time are the pulse propagation and processing times which limit the counting rate for a given wire. Thus, MWPCs are subject to a severe count-rate limitation. A second limitation of MWPCs has been their large pixel size and the relatively small number of pixels across the detector face, as well as parallax effects. These problems have been addressed by changes in the detector geometry (*e.g.* spherical drift chambers; Charpak, 1982), by microfabrication on glass substrates of the wires comprising the back plane of the detector, and by dividing the active area into small zones, each of which is read out independently. Robustness of MWPCs has also been a problem.

The dead time can be reduced by reducing the thickness of the detector. However, reducing the detector thickness reduces the X-ray stopping power. Increasing the gas pressure not only improves the quantum efficiency, but also helps to reduce the dead time further. Unfortunately, high gas pressure complicates the design of the front window of the detector. Despite these problems, two-dimensional gas-detector prototype modules with 200 µm square pixels have been constructed that are expected to have a local linear count-rate limit of $7\ \mathrm{MHz\ mm^{-2}}$ and a quantum efficiency above 80% at energies used in crystallography (see Sarvestani *et al.*, 1998).

7.1.3.2.2. *Integrating detectors*

X-ray film was the first area detector and has a long history of important contributions to the solution of structures and to X-ray imaging (Arndt *et al.*, 1977). In many applications, film has effectively been displaced because of its relative insensitivity caused by the high level of background fog, its multistep processing leading to long delays before digital data are obtained and its nonlinearity. However, X-ray film is still a superior integrator for long exposures, and lithographic film has much higher spatial resolution than any other area detector. Polaroid film in an X-ray cassette is an excellent diagnostic tool for beam alignment problems. And, in all cases, film is inexpensive.

Storage phosphors, also called image plates, are probably the most widely used area X-ray detector for crystallography at present, particularly in laboratories with conventional X-ray sources (Amemiya *et al.*, 1988; Eikenberry *et al.*, 1992). These sheets of material are a much improved functional replacement for X-ray film in many applications, including medical radiography and autoradiography in biological research. Storage phosphors are made from a BaFBr:Eu or other photostimulable phosphor coated on a suitable backing. These phosphors have the property that absorbed X-ray energy can be trapped in long-lived states within the phosphor grains, and that this energy can later be released as blue fluorescence upon photostimulation with red light. Grain-size distribution, grain orientation, binder choice and coating thickness are important parameters in the commercial preparation of the sheets.

The exposed phosphor sheet is raster-scanned with a finely focused red laser and the resulting photostimulated emission is recorded by a photomultiplier. The result is a digital image of the X-ray intensity distribution. The scanning can be done either on-line in self-contained systems or off-line in a separate scanning instrument. Scanning typically requires several minutes. In advanced scanners, this is reduced to several tens of seconds, where the limit is set by the time constant of the photostimulated emission process, which in turn determines the minimum time the laser should dwell on each pixel. The off-line scanner, preferred at synchrotron sources, permits a new exposure to be made while scanning is performed. Self-contained systems offer the advantages of simplified operation and the possibility of calibrating the detector, since only a single sheet of material is used in a mechanically stable setup.

Storage phosphors are typically read out with 100 µm square pixels, resulting in 2000×2500 pixel images for the common size of sheet. Larger formats are available. The wide PSF of storage phosphors makes the effective pixel size considerably larger than the nominal value. Some scanners permit smaller pixels, but this is of limited utility because there is a readout noise component associated with each pixel and too small a pixel harms the signal-to-noise ratio without improving data. The most critical component of the storage-phosphor system is the mirror assembly that gathers the photostimulated emission during readout. There are very few emitted photons for each stored X-ray (at the photon energies used for diffraction), and only a small fraction of these are detected in the photomultiplier (in some cases less than one per stored X-ray). This step in the detection process is critical in maintaining high quantum efficiency. Although image plates have an inherently wide dynamic range, the practical value is always limited by the scanner analogue-to-digital converter.

Television detectors. Numerous integrating detector designs based on television sensor technologies have been published and, in one case, produced commercially (Milch *et al.*, 1982; Arndt, 1991). These detectors span a wide range of design complexity and performance. The primary element is a phosphor screen, which converts the incident X-ray pattern to a light image that is directly or

indirectly coupled to the sensor, such as a Vidicon or CCD. Many of the designs employ image intensifiers to raise the signal strength of the visible image above the noise of the sensor system. Some designs employ cameras operated at video rates, with frames accumulated in the attached computer or on videotape. Other designs use cooled cameras operated in a slow-scan mode, which greatly reduces noise. The X-ray exposure is integrated in the camera, then read out once at the end of the integration period.

Most of these systems would be classified as complex, but several of them are working reliably today in laboratories with conventional X-ray sources. The image intensifiers improve the DQE of the systems, while sacrificing dynamic range and image sharpness. In addition, intensifiers are sensitive to magnetic fields, requiring great care in their use if proper detector calibration is to be maintained.

Considerable enhancement to the television-type detector is made possible by the low-noise imaging capabilities of CCDs, described in Chapter 7.2. In this case, high DQE can be maintained without intensification when the CCD is cooled and read with slow-scan electronics. As such, these detectors are much more robust and have improved imaging qualities.

7.1.4. Future detectors

Commercially available X-ray detectors have evolved from X-ray film and point diffractometry to area gas-proportional counters, to image plates, and now to CCD detectors. Two new X-ray detector technologies are on the horizon. One is based on the large-area amorphous semiconductors and thin-film transistor arrays which are being intensively developed by many large companies for medical radiography (reviewed by Moy, 1999). The radiographic need is to be able to cover very large areas (*e.g.* 0.5 m^2) with a high-spatial-resolution detector that is sensitive to hard X-rays. A number of these detectors are at the moment (1999) poised for introduction, but they are specialized for radiographic applications and are poorly suited for relatively long, low-noise integration of low-energy X-rays. It remains to be seen whether the technology will succeed and whether it can be modified for quantitative crystallographic applications.

A second technology being developed specifically for quantitative X-ray diffraction is based on solid-state pixel array detectors (PADs) (Iles *et al.*, 1996; Datte *et al.*, 1999; Barna *et al.*, 1997; Rossi *et al.*, 1999). In a PAD, X-rays are stopped directly in a semiconductor and the resulting signal is processed by electronics integrated into each pixel. Direct conversion of X-rays into electrical signals in a high-grade semiconductor has many advantages: many signal electrons are produced for each X-ray, and the conversion medium is very linear, has low noise and is well understood. Since each pixel has its own electronics, there is enormous flexibility in performing local signal processing. In principle, PADs have tremendous advantages of sensitivity, flexibility, noise and stability. The challenge will be to make PADs of a size and format useful for crystallography, while still being sufficiently affordable to be commercially viable.

images at different exposure times for each position of the crystal, extensible software would make it easier to set up the experiment. Finally, the software should permit access to all of the readout modes of the detector. For instance, a detector may be capable of rapidly scanning a small region of interest for alignment purposes, or it may be capable of streak-mode operation for certain types of time-resolved experiments. Available CCD detector software for macromolecular applications has room for much improvement. Hopefully, software will continue to undergo rapid development. Standardization is especially needed.

7.2.5. Applications to macromolecular crystallography

Storage rings. CCD detectors have gained widespread acceptance for macromolecular crystallography at storage-ring sources, in part because of the high-quality data they give, but more for their speed, convenience and efficiency. Accurate data to high resolution are especially important for MAD phasing, and CCD detectors excel in this application. In the past with film, or even storage phosphors, teams of perhaps ten people were required to perform a synchrotron experiment; today, a single person per shift can perform an experiment. With increasing beam flux, improved X-ray optics and faster CCDs, it is often possible to collect full data sets in little more than an hour. Anticipated improvements in speed for CCD detectors should soon make it feasible to collect fine-sliced rotation data routinely; these data are expected to yield better structure solutions.

Home laboratories. Acceptance of CCD detectors for macromolecular crystallography at home laboratories has been slower, in part because there is not such a premium on speed, and in part because of cost. Diffracted spot sizes are larger than at synchrotrons, so highly accurate data should be obtainable. Fully automatic storage phosphor systems work quite well with conventional sources and at this time are lower in cost than large CCD detectors. However, they have a minimum cycle time, caused by the mechanics of the readout scheme, and the required exposure for a strongly diffracting crystal can best this time by a wide margin. Thus, for strongly diffracting specimens, CCD detectors can be significantly more efficient.

7.2.6. Future of CCD detectors

The basic principles of CCD detector technology are now well developed, but various incremental improvements have already been demonstrated and may be expected in commercial detectors. These include larger detector areas, faster read times (owing to both faster electronics and multiamplifier CCDs), more flexible control electronics, better optimized phosphors and calibrations, and, especially, better software. Lower-cost CCD detectors would certainly be welcome. It is easily predicted that the application of CCD detectors will continue to increase rapidly for at least several more years until displaced by even better technologies, such as pixel array detectors (see Section 7.1.4 in Chapter 7.1).

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7.1

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