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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

references