11. DATA PROCESSING

11.1. Automatic indexing of oscillation images

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11.1.1. Introduction

Auto-indexing routines have been used extensively for initiating diffraction data collection with a single-point-detector device (Sparks, 1976, 1982). These methods depend upon the precise knowledge of the reciprocal-lattice vectors for a few selected reflections. Greater difficulty has been encountered for automatic indexing of oscillation images recorded on two-dimensional detectors using randomly oriented crystals, as is frequently the case for macromolecular crystal samples. In the past, the practice was to orient crystals relative to the camera axes with an accuracy of at least 1°. In this case, the indexing procedure required only refinement of the crystal orientation matrix (Wonacott, 1977; Rossmann, 1979). The 'American method' (Rossmann & Erickson, 1983), where crystals are oriented more or less randomly, is currently used because of the need for optimizing available synchrotron time and because of the deterioration in radiation-sensitive crystals during the setting process.

A variety of techniques were suggested to determine the crystal orientation, some of which required initial knowledge of the cell dimensions (Vriend & Rossmann, 1987; Kabsch, 1988), while more advanced techniques (Kim, 1989; Higashi, 1990; Kabsch, 1993) determined both cell dimensions and crystal orientation. All these methods start with the determination of the reciprocal-lattice vectors assuming that the oscillation photographs are 'stills'. The methods of Higashi and Kabsch, as well as, in part, Kim's, analyse the distribution of the difference vectors generated from the reciprocal-lattice vectors. The most frequent difference vectors are taken as the basis vectors defining the reciprocal-lattice unit cell and its orientation. In addition, Kim's technique requires the input of the orientation of a likely zone-axis direction onto which the reciprocal-lattice vectors are then projected. The projections will have a periodicity distribution consistent with the reciprocal-lattice planes perpendicular to the zone axis. Duisenberg (1992) used a similar approach for single-point-detector data, although he did not rely on prior knowledge of the zone-axis direction. Instead, he defined possible zone axes as being perpendicular to a reciprocal-lattice plane by combining three, suitably chosen, reciprocal-lattice points.

None of the above techniques were entirely satisfactory as they sometimes failed to find a suitable crystal orientation matrix. A major advance was made in the program *DENZO*, a part of the *HKL* package (Otwinowski & Minor, 1997), which not only has a robust indexing procedure but also has a useful graphical interface. Unfortunately, the indexing technique used in the procedure has never been described, except for a few hints in the manual on the use of an FFT (fast Fourier transform). Indeed, Bricogne (1986) suggested that a three-dimensional Fourier transformation might be a powerful indexing tool, and Strouse (1996) developed such a procedure for single-point-detector data. However, for large unit cells this procedure requires an excessive amount of memory and time (Campbell, 1997).

11.1.2. The crystal orientation matrix

The position $\mathbf{x}$ (x, y, z) of a reciprocal-lattice point can be given as

$$\mathbf{x} = [\Phi][A]\mathbf{h}. \qquad (11.1.2.1)$$

The matrix $[\Phi]$ is a rotation matrix around the camera's spindle axis for a rotation of φ. The vector $\mathbf{h}$ represents the Miller indices (h, k, l) and $[A]$ defines the reciprocal unit-cell dimensions and the orientation of the crystal lattice with respect to the camera axes when $\varphi = 0$. Thus,

$$[A] = \begin{pmatrix} a_x^* & b_x^* & c_x^* \\ a_y^* & b_y^* & c_y^* \\ a_z^* & b_z^* & c_z^* \end{pmatrix}, \qquad (11.1.2.2)$$

where a_x^*, a_y^* and a_z^* are the components of the crystal $\mathbf{a}^*$ axis with respect to the orthogonal camera axes. When an oscillation image is recorded, the position of a reciprocal-lattice point is moved from $\mathbf{x}_1$ to $\mathbf{x}_2$, corresponding to a rotation of the crystal from φ_1 to φ_2. The recorded position of the reflection on the detector corresponds to the point $\mathbf{x}$ when it is on the Ewald sphere somewhere between $\mathbf{x}_1$ and $\mathbf{x}_2$. The actual value of φ at which this crossing occurs cannot be retrieved from the oscillation image. We shall therefore assume here, as is the case in all other procedures, that $[\Phi][A]$ defines the crystal orientation in the centre of the oscillation range. Defining the camera axes as in Rossmann (1979), it is easy to show that a reflection recorded at the position (X, Y) on a flat detector normal to the X-ray beam, at a distance D from the crystal, corresponds to

$$\begin{aligned} x &= \frac{X}{\lambda(X^2 + Y^2 + D^2)^{1/2}} \\ y &= \frac{Y}{\lambda(X^2 + Y^2 + D^2)^{1/2}} \\ z &= \frac{D}{\lambda(X^2 + Y^2 + D^2)^{1/2}}, \end{aligned} \qquad (11.1.2.3)$$

where λ is the X-ray wavelength.

If an approximate $[A]$ matrix is available, the Miller indices of an observed peak at (X, Y) can be roughly determined using (11.1.2.3) and (11.1.2.1), where

$$\mathbf{h} = [A]^{-1}[\Phi]^{-1}\mathbf{x}, \qquad (11.1.2.4)$$

with the error being dependent upon the width of the oscillation range, the error in the detector parameters and errors in determining the coordinates of the centres of the recorded reflections.

11.1.3. Fourier analysis of the reciprocal-lattice vector distribution when projected onto a chosen direction

If the members of a set of reciprocal-lattice planes perpendicular to a chosen direction are well separated, then the projections of the reciprocal-lattice vectors onto this direction will have an easily recognizable periodic distribution (Fig. 11.1.3.1). Unlike the procedure of Kim (1989), which requires the input of a likely zone-axis direction, the present procedure tests all possible directions and analyses the frequency distribution of the projected reciprocal-lattice vectors in each case. Also, unlike the procedure of Kim, the periodicity is determined using an FFT.

references