less than Δ so as to optimize the signal-to-noise ratio of the measurement and to sample the rocking-width profile.

The value of Δ, the crystal rocking width for a given *hkl*, depends on the reciprocal-lattice coordinates of the *hkl* relp (see Section 2.2.7). In the region close to the rotation axis, Δ is large.

In the introductory remarks to the monochromatic methods used, it has already been noted that originally the rotation method involved 360° rotations contributing to the diffraction image. Spot overlap led to loss of reflection data and encouraged Bernal and Weissenberg to devise improvements. With modern synchrotron techniques, the restriction on $\Delta\varphi_{\max}$ (equation 2.2.3.30) can be relaxed for special applications. For example, since the spot overlap that is to be avoided involves relp's from adjacent reciprocal-lattice planes, the different Miller indices *hkl* and $h + l$, k, l do lead in fact to a small difference in Bragg angle. With good enough collimation, a small spot size exists at the detector plane so that the two spots can be resolved. For a standard-sized detector, this is practical for low-resolution data recording. This can be a useful complement to the Laue method where the low-resolution data are rather sparsely stimulated and also tend to occur in multiple Laue spots. Alternatively, a much larger detector can be contemplated and even medium-resolution data can be recorded without major overlap problems. These techniques are useful in some time-resolved applications. For a discussion see Weisgerber & Helliwell (1993). For regular data collection, however, narrow angular ranges are still generally preferred so as to reduce the background noise in the diffraction images and also to avoid loss of any data because of spot overlap.

2.2.3.5. *Blind region*

In normal-beam geometry, any relp lying close to the rotation axis will not be stimulated at all. This situation is shown in Fig. 2.2.3.4. The blind region has a radius of

$$\xi_{\min} = d^*_{\max} \sin\theta_{\max} = \frac{\lambda^2}{2d^2_{\min}}, \qquad (2.2.3.31)$$

and is therefore strongly dependent on $d_{\min}$ but can be ameliorated by use of a short λ. Shorter λ makes the Ewald sphere have a larger radius, *i.e.* its surface moves closer to the rotation axis. At Cu $K\alpha$ for 2 Å resolution, approximately 5% of the data lie in the blind region according to this simple geometrical model. However, taking account of the rocking width Δ, a greater percentage of the data than this is not fully sampled except over very large angular ranges. The actual increase in the blind-region volume due to this effect is minimized by use of a collimated beam and a narrow spectral spread (*i.e.* finely monochromatized, synchrotron radiation) if the crystal is not too mosaic.

These effects are directly related to the Lorentz factor,

$$L = 1/(\sin^2 2\theta - \zeta^2)^{1/2}. \qquad (2.2.3.32)$$

It is inadvisable to measure a reflection intensity when L is large because different parts of a spot would need a different Lorentz factor.

The blind region can be filled in by a rotation about another axis. The total angular range that is needed to sample the blind region is $2\theta_{\max}$ in the absence of any symmetry or $\theta_{\max}$ in the case of *mm* symmetry (for example).

2.2.4. Weissenberg geometry

Weissenberg geometry (Weissenberg, 1924) is dealt with in the books by Buerger (1942) and Woolfson (1970), for example.

2.2.4.1. *General*

The conventional Weissenberg method uses a moving film in conjunction with the rotation of the crystal and a layer-line screen. This allows:

(*a*) A larger rotation range of the crystal to be used (say 200°), avoiding the problem of overlap of reflections (referred to in Subsection 2.2.3.4 on oscillation photography).

(*b*) Indexing of reflections on the photograph to be made by inspection.

The Weissenberg method is not widely used now. In small-molecule crystallography, quantitative data collection is usually performed by means of a diffractometer.

Weissenberg geometry has been revived as a method for macromolecular data collection (Sakabe, 1983, 1991), exploiting monochromatized synchrotron radiation and the image plate as detector. Here the method is used without a layer-line screen where the total rotation angle is limited to $\sim 15°$; this is a significant increase over the rotation method with a stationary film. The use of this effectively avoids the presence of partial reflections and reduces the total number of exposures required. Provided the Weissenberg camera has a large radius, the X-ray background accumulated over a single spot is actually not serious. This is because the X-ray background decreases approximately according to the inverse square of the distance from the crystal to the detector.

The following Subsections 2.2.4.2 and 2.2.4.3 describe the standard situation where a layer-line screen is used.

2.2.4.2. *Recording of zero layer*

Normal-beam geometry (*i.e.* the X-ray beam perpendicular to the rotation axis) is used to record zero-layer photographs. The film is held in a cylindrical cassette coaxial with the rotation axis. The centre of the gap in a screen is set to coincide with the zero-layer plane. The coordinate of a spot on the film measured parallel (Z_F) and perpendicular (Y_F) to the rotation axis is given by

$$Y_F = \frac{2\pi}{360} D\Upsilon \qquad (2.2.4.1)$$

$$Z_F = \varphi/f, \qquad (2.2.4.2)$$

where φ is the rotation angle of the crystal from its initial setting, f is the coupling constant, which is the ratio of the crystal rotation angle divided by the film cassette translation distance, in $^\circ\text{min}^{-1}$, and D is the camera radius. Generally, the values of f and D are $2^\circ\ \text{min}^{-1}$ and 28.65 mm, respectively.

2.2.4.3. *Recording of upper layers*

Upper-layer photographs are usually recorded in equi-inclination geometry [*i.e.* $\mu = -\nu$ in equations (2.2.3.7) and (2.2.3.8)]. The X-ray-beam direction is made coincident with the generator of the cone of the diffracted beam for the layer concerned, so that the incident and diffracted beams make equal angles (μ) with the equatorial plane, where

$$\mu = \sin^{-1}\zeta_n/2. \qquad (2.2.4.3)$$

The screen has to be moved by an amount

$$s\tan\mu, \qquad (2.2.4.4)$$

where s is the screen radius. If the cassette is held in the same position as the zero-layer photograph, then reflections produced by the same orientation of the crystal will be displaced

$$D\tan\mu \qquad (2.2.4.5)$$

Table 2.2.5.1. *The distance displacement (in mm) measured on the film versus angular setting error of the crystal for a screenless precession ($\bar{\mu} = 5^\circ$) setting photograph*

Angular correction, ε, in degrees and minutes	Δ r.l.u	Distance displacement (mm) for three crystal-to-film distances		
		60 mm	75 mm	100 mm
0	0	0	0	0
15′	0.0175	1.1	1.3	1.8
30′	0.035	2.1	2.6	3.5
45′	0.0526	3.2	4.0	5.3
60′	0.070	4.2	5.3	7.0
1° 15′	0.087	5.2	6.5	8.7
1° 30′	0.105	6.3	7.9	10.5
1° 45′	0.123	7.4	9.2	12.3
2°	0.140	8.4	10.5	14.0

Alternatively, $\Delta = \delta/D \simeq \sin 4\varepsilon$ can be used if ε is small [from equation (2.2.5.1)].

Notes

(1) A value of $\bar{\mu}$ of 5° is assumed although there is a negligible variation in ε with $\bar{\mu}$ between 3° (typical for proteins) and 7° (typical for small molecules).

(2) Crystal-to-film distances on a precession camera are usually settable at the fixed distance $D = 60$, 75, and 100 mm.

(3) This table should be used in conjunction with Fig. 2.2.5.1.

(4) Values of ε are given in intervals of 5′ as this is convenient for various goniometer heads which usually have verniers in 5′, 6′ or 10′ units. The vernier on the spindle of the precession camera is often in 2′ units.

relative to the zero-layer photograph. This effect can be eliminated by initial translation of the cassette by $D \tan \mu$.

2.2.5. Precession geometry

The main book dealing with the precession method is that of Buerger (1964).

2.2.5.1. *General*

The precession method is used to record an undistorted representation of a single plane of relp's and their associated intensities. In order to achieve this, the crystal is carefully set so that the plane of relp's is perpendicular to the X-ray beam. The normal to this plane, the zone axis, is then precessed about the X-ray-beam axis. A layer-line screen allows only relp's of the plane of interest to pass through to the film. The motion of the crystal, screen, and film are coupled together to maintain the coplanarity of the film, screen, and zone.

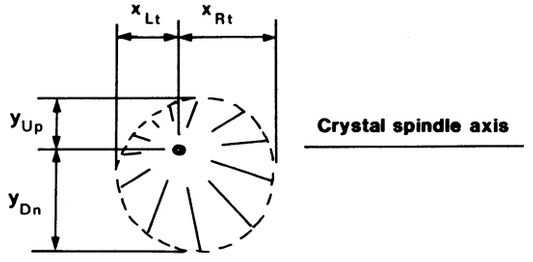

Fig. 2.2.5.1. The screenless precession setting photograph (schematic) and associated mis-setting angles for a typical orientation error when the crystal has been set previously by a monochromatic still or Laue.

2.2.5.2. *Crystal setting*

Setting of the crystal for one zone is carried out in two stages. First, a Laue photograph is used for small molecules or a monochromatic still for macromolecules to identify the required zone axis and place it parallel to the X-ray beam. This is done by adjustment to the camera-spindle angle and the goniometer-head arc in the horizontal plane. This procedure is usually accurate to a degree or so. Note that the vertical arc will only rotate the pattern around the X-ray beam. Second, a screenless precession photograph is taken using an angle of ~ 7–10° for small molecules or 2–3° for macromolecules. It is better to use unfiltered radiation, as then the edge of the zero-layer circle is easily visible. Let the difference of the distances from the centre of the pattern to the opposite edges of the trace in the direction of displacement be called $\delta = D\Delta$ so that for the horizontal goniometer-head arc and the dial: $\delta_{\rm arc} = x_{\rm Rt} - x_{\rm Lt}$ and $\delta_{\rm dial} = y_{\rm Up} - y_{\rm Dn}$ (Fig. 2.2.5.1). The corrections ε to the arc and camera spindle are given by

$$\Delta = \frac{\delta}{D} = \frac{\sin 4\varepsilon \cos \bar{\mu}}{\cos^2 2\varepsilon - \sin^2 \bar{\mu}} \text{ in r.l.u.,} \qquad (2.2.5.1)$$

where D is the crystal-to-film distance and $\bar{\mu}$ is the precession angle.

It is possible to measure δ to about 0.3 mm ($\delta = 1$ mm corresponds to 14′ error for $D = 60$ mm and $\bar{\mu} \simeq 7^\circ$ [Table 2.2.5.1, based on *IT* II (1959, p. 200)].

2.2.5.3. *Recording of zero-layer photograph*

Before the zero-layer photograph is taken, an Nb filter (for Mo $K\alpha$) or an Ni filter (for Cu $K\alpha$) is introduced into the X-ray beam path and a screen is placed between the crystal and the film at a distance from the crystal of

$$s = r_s \cot \bar{\mu}, \qquad (2.2.5.2)$$

where r_s is the screen radius. Typical values of $\bar{\mu}$ would be 20° for a small molecule with Mo $K\alpha$ and 12–15° for a protein with Cu $K\alpha$. The annulus width in the screen is chosen usually as 2–3 mm for a small molecule and 1–2 mm for a macromolecule. A clutch slip allows the camera motor to be disengaged and the precession motion can be executed under hand control to check for fouling of the goniometer head, crystal, screen or film cassette; s and r_s need to be selected so as to avoid this happening. The zero-layer precession photograph produced has a radius of $2D \sin \bar{\mu}$ corresponding to a resolution limit $d_{\rm min} = \lambda/2 \sin \bar{\mu}$. The distance between spots A is related to the reciprocal-cell parameter a^* by the formula

$$a^* = \frac{A}{D}. \qquad (2.2.5.3)$$

2.2.5.4. *Recording of upper-layer photographs*

The recording of upper-layer photographs involves isolating the net of relp's at a distance from the zero layer of $\zeta_n = n\lambda/b$, where b is the case of the b axis antiparallel to the X-ray beam. In order to determine ζ_n, it is generally necessary to record a cone-axis photograph. If the cell parameters are known, then the camera settings for the upper-level photograph can be calculated directly without the need for a cone-axis photograph.

In the upper-layer precession photograph, the film is advanced towards the crystal by a distance

$$D\zeta_n \qquad (2.2.5.4)$$

and the screen is placed at a distance

references