

9. MONOCHROMATIC DATA COLLECTION

Table 9.1.7.2. *Rotation range (°) required in different crystal classes*

The direction of the spindle axis is given in parentheses; *ac* means any vector in the *ac* plane.

Point group	Native data	Anomalous data
1	180 (any)	180 + 2θ _{max} (any)
2	180 (<i>b</i>); 90 (<i>ac</i>)	180 (<i>b</i>); 180 + 2θ _{max} (<i>ac</i>)
222	90 (<i>ab</i> or <i>ac</i> or <i>bc</i>)	90 (<i>ab</i> or <i>ac</i> or <i>bc</i>)
4	90 (<i>c</i> or <i>ab</i>)	90 (<i>c</i>); 90 + θ _{max} (<i>ab</i>)
422	45 (<i>c</i>); 90 (<i>ab</i>)	45 (<i>c</i>); 90 (<i>ab</i>)
3	60 (<i>c</i>); 90 (<i>ab</i>)	60 + 2θ _{max} (<i>c</i>); 90 + θ _{max} (<i>ab</i>)
32	30 (<i>c</i>); 90 (<i>ab</i>)	30 + θ _{max} (<i>c</i>); 90 (<i>ab</i>)
6	60 (<i>c</i>); 90 (<i>ab</i>)	60 (<i>c</i>); 90 + θ _{max} (<i>ab</i>)
622	30 (<i>c</i>); 90 (<i>ab</i>)	30 (<i>c</i>); 90 (<i>ab</i>)
23	~60	~70
432	~35	~45

required to estimate the effective completeness and derive optimum strategies. For minimalist approaches to obtaining a high completeness, the importance of selecting the total rotation range, the optimal starting point and indeed the crystal orientation must be stressed. This means that the crystal orientation must be defined at the start of the experiment from the initial exposures.

9.1.7.2. *Total rotation range for anomalous-dispersion data*

In the presence of anomalous-scattering centres, Friedel's law breaks down and the intensities of the two halves of the reciprocal sphere are no longer equivalent. Strictly speaking, reflections related by a centre of symmetry or mirror relation cease to have equal intensities, but those related by pure rotation preserve their equivalence. The non-equivalent pairs of reflections are known as Bijvoet pairs. In macromolecular crystallography, it is often highly desirable to record the intensity differences between the Bijvoet mates to provide information on the position of anomalous scatterers, usually to be exploited in phasing procedures (Part 14). The anomalous signal should also be retained for so-called native data, for example, in the discrimination between water and ions in the surface solvent shell.

This implies that the intensities of the unique reflections have to be measured for both hemispheres of reciprocal space. In the general (triclinic) case, this requires the rotation of the crystal by a wider rotation range. At very low resolution, the surface of the Ewald sphere can be approximated by a plane. In this case, rotation of the lower half of the Ewald sphere will cover a full hemisphere of data, and the upper half the remaining centrosymmetrically related hemisphere. At high resolution, the surface of the Ewald sphere increasingly deviates from planarity by θ on each side (Fig. 9.1.7.6). To record complete anomalous data for such a triclinic crystal therefore requires it to be rotated by $180^\circ + 2\theta_{\max}$ from a random starting position. This will measure each Bijvoet mate at least once. However, only after a total rotation of 360° will the average multiplicity reach a value of two.

Similar reasoning applies to higher-symmetry space groups. Intensity data for two asymmetric units related by a centre of symmetry or a mirror need to be recorded. For some cases, the total range remains the same for completeness of anomalous data as for native. However, in several symmetries or orientations, the total range must again be increased by either θ_{\max} or $2\theta_{\max}$ (Table 9.1.7.2).

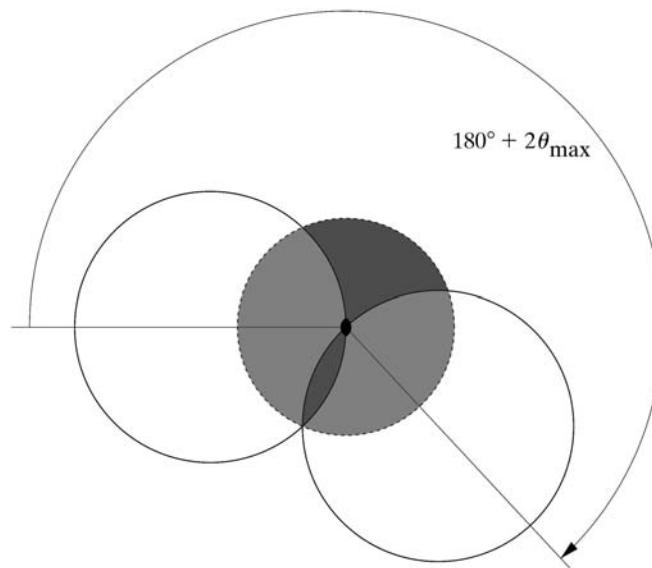


Fig. 9.1.7.6. For data containing an anomalous signal, when both Bijvoet mates have to be measured, 180° rotation of a triclinic crystal is not sufficient and at least an additional $2\theta_{\max}$ is required.

9.1.7.3. *Blind region*

Even after rotation of the crystal about a single axis by 360° , some reflections do not cross the surface of the Ewald sphere and cannot be measured. These lie in a cusp around the rotation axis which is referred to as the blind region. This is in principle a disadvantage of the single-rotation method, but for most systems the problems are easily overcome. Owing to the curvature of the Ewald sphere, the width of the blind region increases with the resolution and directly depends on a single parameter, the diffraction angle θ (Fig. 9.1.7.7). The variation of the fraction, B_θ , of unrecordable reflections lying in the blind region at a particular resolution with Bragg angle θ is given by

$$B_\theta = 1 - \cos \theta.$$

The cumulative fraction, B_{tot} , of reflections in the blind region up to a certain resolution is given by

$$B_{\text{tot}} = 1 - 3(4\theta - \sin 4\theta)/(32 \sin^3 \theta).$$

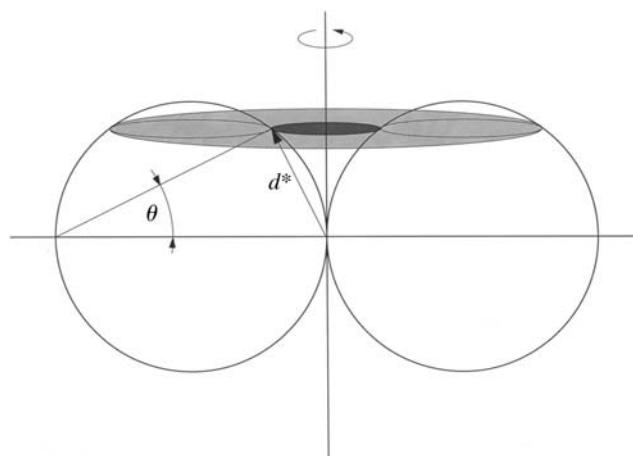


Fig. 9.1.7.7. Rotation by 360° leaves the part of the reciprocal space in the blind region unmeasured, since the reflections near the rotation axis do not cross the surface of the Ewald sphere. The rotation axis in this projection lies vertically in the plane of the figure.