

2. DIFFRACTION GEOMETRY AND ITS PRACTICAL REALIZATION

$$s_n = r_s \cot \bar{v}_n = r_s \cot \cos^{-1}(\cos \bar{\mu} - \zeta_n). \quad (2.2.5.5)$$

The resulting upper-layer photograph has outer radius

$$D(\sin \bar{v}_n + \sin \bar{\mu}) \quad (2.2.5.6)$$

and an inner blind region of radius

$$D(\sin \bar{v}_n - \sin \bar{\mu}). \quad (2.2.5.7)$$

2.2.5.5. Recording of cone-axis photograph

A cone-axis photograph is recorded by placing a film enclosed in a light-tight envelope in the screen holder and using a small precession angle, *e.g.* 5° for a small molecule or 1° for a protein. The photograph has the appearance of concentric circles centred on the origin of reciprocal space, provided the crystal is perfectly aligned. The radius of each circle is

$$r_n = s \tan \bar{v}_n, \quad (2.2.5.8)$$

where

$$\cos \bar{v}_n = \cos \bar{\mu} - \zeta_n. \quad (2.2.5.9)$$

Hence, $\zeta_n = \cos \bar{\mu} - \cos \tan^{-1}(r_n/s)$.

2.2.6. Diffractometry

The main book dealing with single-crystal diffractometry is that of Arndt & Willis (1966). Hamilton (1974) gives a detailed treatment of angle settings for four-circle diffractometers. For details of area-detector diffractometry, see Howard, Nielsen & Xuong (1985) and Hamlin (1985).

2.2.6.1. General

In this section, we describe the following related diffractometer configurations:

(a) normal-beam equatorial geometry [ω, χ, φ option or ω, κ, φ (kappa) option];

(b) fixed $\chi = 45^\circ$ geometry with area detector.

(a) is used with single-counter detectors. The kappa option is also used in the television area-detector system of Enraf-Nonius (the FAST). (b) is used with the multiwire proportional chamber, XENTRONICS, system. (FAST is a trade name of Enraf-Nonius; XENTRONICS is a trade name of Siemens.)

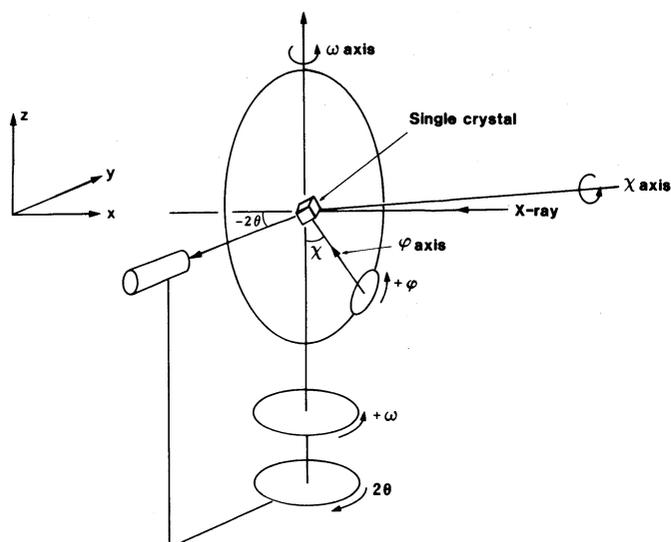


Fig. 2.2.6.1 Normal-beam equatorial geometry: the angles $\omega, \chi, \varphi, 2\theta$ are drawn in the convention of Hamilton (1974).

The purpose of the diffractometer goniostat is to bring a selected reflected beam into the detector aperture or a number of reflected beams onto an area detector of limited aperture (*i.e.* an aperture that does not intercept all the available diffraction spots at one setting of the area detector) [see Hamlin (1985, p. 431), for example].

Since the use of electronic area detectors is now increasingly common, the properties of these detectors that relate to the geometric prediction of spot position will be described later.

The single-counter diffractometer is primarily used for small-molecule crystallography. In macromolecular crystallography, many refl's are almost simultaneously in the diffraction condition. The single-counter diffractometer was extended to five separate counters [for a review, see Artymiuk & Phillips (1985)], then subsequently to a multi-element linear detector [for a review, see Wlodawer (1985)]. Area detectors offer an even larger aperture for simultaneous acquisition of reflections [Hamlin *et al.* (1981), and references therein].

Large-area on-line image-plate systems are now available commercially to crystallographers, whereby the problem of the limited aperture of electronic area detectors is circumvented and the need for a goniostat is relaxed so that a single axis of rotation can be used. Systems like the R-AXISIIc (Rigaku Corporation) and the MAR (MAR Research Systems) fall into this category, utilizing IP technology and an on-line scanner. A next generation of device beckons, involving CCD area detectors. These offer a much faster duty cycle and greater sensitivity than IP's. By tiling CCD's together, a larger-area device can be realized. However, it is likely that these will be used in conjunction with a three-axis goniostat again, except in special cases where a complete area coverage can be realized.

2.2.6.2. Normal-beam equatorial geometry

In normal-beam equatorial geometry (Fig. 2.2.6.1), the crystal is oriented specifically so as to bring the incident and reflected beams, for a given refl, into the equatorial plane. In this way, the detector is moved to intercept the reflected beam by a single rotation movement about a vertical axis (the 2θ axis). The value of θ is given by Bragg's law as $\sin^{-1}(d^*/2)$. In order to bring \mathbf{d}^* into the equatorial plane (*i.e.* the Bragg plane into the meridional plane), suitable angular settings of a three-axis goniostat are necessary. The convention for the sign of the angles given in Fig. 2.2.6.1 is that of Hamilton (1974); his choice of sign of 2θ is adhered to despite the fact that it is left-handed, but in any case the signs of ω, χ, φ are standard right-handed. The

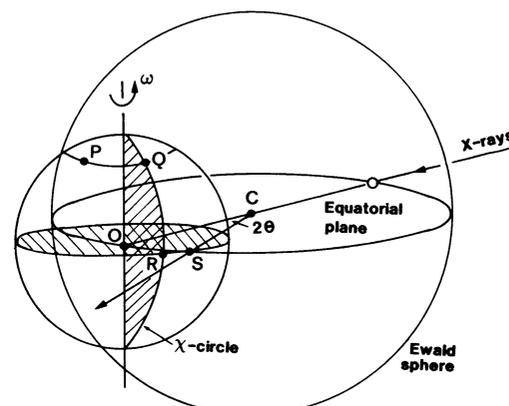


Fig. 2.2.6.2. Diffractometry with normal-beam equatorial geometry and angular motions ω, χ and φ . The refl at P is moved to Q via φ , from Q to R via χ , and R to S via ω . From Arndt & Willis (1966). In this specific example, the φ axis is parallel to the ω axis (*i.e.* $\chi = 0^\circ$).