2. Reflexion 'on the corner'

In this case the incident rays fall on one face and the diffracted rays leave through the adjacent face. Only the region at the common crystal edge makes a contribution to the absorption factor. The above integral for this case gives the formula

$$A_\delta = \frac{1}{S}\frac{\sin\psi_1 \sin\psi_2}{\mu^2 \sin\delta},$$

ψ_1 and ψ_2 being now the angles made by the diffracted and incident rays, respectively, with the appropriate crystal faces (e.g. for the corner at B this formula is to be applied when the angle ψ_1 of the diffracted ray with BC is greater than 76°; ψ_2 is then the angle of the incident ray with AB). In the case of mercury diphenyl, δ has only the value δ_1 or δ_2. It may be noted that the formula is no longer accurate when ψ_2 and φ_2 are nearly zero.

The above formulae are applicable to the zero-layer reflexions of any prism-shaped crystal of great absorbing power. They were applied successfully in the structure analysis (soon to be published) of mercury diethylene oxide (μ = 550 cm.$^{-1}$ for Cu $K\alpha$ radiation), the crystal of which had a hexagonal cross section perpendicular to the needle axis.

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Determination of interaxial angles of a triclinic crystal from a single setting.

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Buerger (1942) has described how all parameters of the crystal lattice may be determined from a single setting of a triclinic crystal on a Weissenberg goniometer. This communication presents a simplified method for measuring the angles α and β (the axis of rotation is taken as c). Buerger suggests two methods for measuring these angles: the first (p. 377) depends on measuring the height above the film center line of the bottom of the festoon $h01$ for α (and $0k1$ for β); the second (p. 383) requires a specially prepared photograph, the necessity for which is often not evident until the first layer has been recorded. The procedure described below is more accurate than the first and, unlike the second, is applied to the normal record of the first layer.

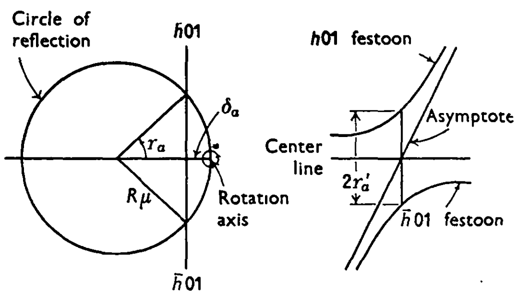

Fig. 1. Left: The intersection of the sphere of reflection with the first layer of the reciprocal lattice. Right: The corresponding section of a Weissenberg photograph.

As in Buerger's procedure, we require the perpendicular displacement δ_α of the reciprocal lattice row $h01$ from the intersection of the rotation axis with the first layer (and similarly for β). The left diagram in Fig. 1 illustrates the orientation of the first layer of the reciprocal lattice at the instant it is recorded in the center of the portion of Weissenberg photograph shown on the right. The ($h01$) spots which appear on the photograph enable the $h01$ and $\bar{h}01$ festoons to be drawn in, as well as their common asymptote. The vertical distance $2\Upsilon_\alpha'$ between the festoons is then measured at the intersection of the asymptote with the center line. Then the angle Υ_α (rad.) is equal to Υ_α'/r, where r is the camera radius. For a camera of 57·3 mm. diameter, $\Upsilon_\alpha(°) = 2\Upsilon_\alpha'$ (mm.). The angle α is then calculated from these relations, which follow from Fig. 1 and from Buerger's treatment:

$$\delta_\alpha = R_\mu(1-\cos\Upsilon_\alpha),$$

where $R_\mu = (\cos\mu)/\lambda$ is the radius of the circle of reflection in the layer with equi-inclination angle μ;

$$\tan\alpha = -\zeta/\delta_\alpha;$$
$$\zeta = 1/c.$$

Geometrical consideration of the effect of various errors yields the following results. A 1° error in mis-setting the equi-inclination angle μ or in aligning the c axis with the axis of rotation causes no more than a $\frac{1}{2}°$ error in the value of α, provided $c/\lambda \leq 10$. Since the shortest axis is usually chosen to rotate the crystal about, this requirement is usually satisfied. A 2° error in locating the point of intersection of the asymptote with the center line (corresponding to 1 mm. along the center line on the 57·3 mm. diameter camera) causes no more than a $\frac{1}{3}°$ error in the value of α, provided $c/\lambda \leq 10$. A 1° error in Υ_α (corresponding to a 1 mm. error in the measurement of $2\Upsilon_\alpha'$ with the 57·3 mm. diameter camera) causes no more than a 2·5° error in α, provided $c/\lambda \leq 10$.

Reference

BUERGER, M. J. (1942). *X-Ray Crystallography*. New York: Wiley.