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A Method for Correcting Equi-inclination Weissenberg Settings

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A new method for correcting equi-inclination Weissenberg camera settings is proposed. The method is based on the observation that if an incorrect value for the equi-inclination angle is used the movement of the layer line screen required, for the diffracted beams to pass through the slot, is not that calculated. Examples are given to show that the accuracy can be high in favourable cases and moderately high in unfavourable circumstances. Compared with Sayre's method, this technique has two advantages in that it is perfectly general and there is no need to take Weissenberg photographs before applying corrections.

Equi-inclination Weissenberg camera settings are calculated from the lattice spacing parallel to the rotation axis of the crystal, and if the lattice spacing is not accurately known the equi-inclination settings will themselves be in error. It has already been pointed out by Sayre (1954) that an important consequence of this is that the Lorentz factor can be seriously in error, especially for near-in spots.

In addition, the use of incorrect settings will cause layer lines which should pass through the rotation axis not to intersect when upper layer photographs are taken. This in turn will cause layer lines which should be straight on upper layer photographs to become curved. Sayre (1954) has already devised a method for obtaining corrections to be applied to the equi-inclination angle μ by considering the curvature of these lines.

Unfortunately, there are several disadvantages of Sayre's method. It is not a general method; it will not work for crystals which do not give straight layer lines on upper layer photographs, *i.e.* in general triclinic crystals and monoclinic ones where the axis of rotation is not the unique one. In addition, before the method can be applied, one must have already taken a Weissenberg photograph of at least part of the upper layer before being able to apply corrections. Work which has taken some considerable time may have to be repeated, especially if a 180° Weissenberg photograph has been taken, either because missetting is not suspect or because the position of straight layer lines is unknown. Also the spots furthest from the straight layer line are those which are closest to the rotation axis and so may be elongated owing to strain or, on one half of the film, because of Phillips's (1954) effect; measurement of these spots may be inaccurate.

However, in practice it is found that if a slightly inaccurate equi-inclination angle is set, then the calculated movement of the layer line screen for the diffracted beams to pass through the centre of the slot will not agree with that observed. It is found that the difference between the calculated and observed values increases with increasing equi-inclination angle and if the angle used is larger than the correct one the observed movement of the layer line screen is less than the calculated one to receive the diffracted beams. The method proposed is based on these observations.

Consider the geometry of the equi-inclination technique using a correct value of the layer coordinate ζ_{cor} (Fig. 1): then we have for equi-inclination angle

$$\sin \mu_{cor} = \zeta_{cor}/2$$

and for layer line screen movement,

$$s_{cor} = R \tan \mu_{cor} ,$$

where R is the radius of the layer line screen.

Now assume that the value of the layer coordinate is in error, the value calculated from another method being ζ_{calc}. This will give values of

$$\sin \mu_{calc} = \zeta_{calc}/2$$
$$S_{calc} = R \tan \mu_{calc} .$$

But the actual movement of the layer line screen will not be that calculated but will be given by (Fig. 2).

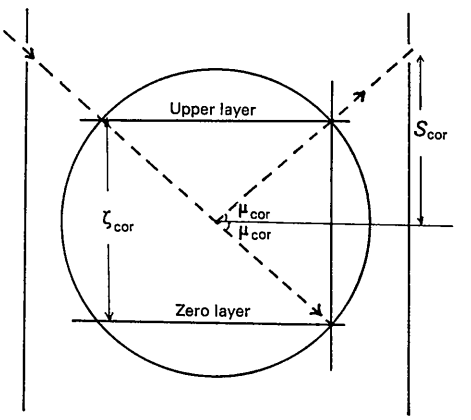

Fig. 1. Geometry of an equi-inclination Weissenberg camera in which the correct settings have been made. View perpendicular to both the beam and the rotation axis.

$$S_{act} = R \tan \delta$$

where $$\sin \delta = \zeta_{cor} - \zeta_{calc}/2 .$$

Thus $$\zeta_{cor} = \sin \delta + \zeta_{calc}/2$$
$$= \sin \delta + \sin \mu_{calc}$$

or $$\sin \mu_{cor} = \tfrac{1}{2}(\sin \delta + \sin \mu_{calc}) .$$

At first sight it might appear that this technique is rather inaccurate in view of the small differences measured but differentiating the correct equation gives:

$$\Delta s = R \sec^2 \mu \Delta \mu + \Delta R \tan \mu .$$

Assuming $R = 24$ mm, $\Delta R = 0$, and $\Delta s = \pm 0{\cdot}05$ mm (*i.e.* the screen positions can be measured to the nearest 0·1 mm) then it is found that $\Delta \mu = \pm 3\frac{1}{2}'$ when $\mu = 45°$ and $\Delta \mu = \pm 7'$ when $\mu = 0°$. Thus the theoretical accuracy is quite high in this case.

On the other hand if $\Delta s = 0$ but there is an error in R, then

$$\Delta \mu = -(\Delta R/R) \sin \mu \cos \mu ,$$

and assuming $\Delta R/R = 0{\cdot}01$, $\Delta \mu = 17'$ when $\mu = 45°$. Thus 1% errors in R cause comparatively large errors in μ when the latter is about 45°. This leads to three important considerations: first ,R must be known to an accuracy better than 1%; fortunately, using micrometer verniers the accuracy can easily be improved to 0·1%. Secondly, the layer line screen must not be distorted from cylindrical, say, by mechanical abuse and, finally, each camera must be considered individually as to the correct value of R to be used. For example, for the Unicam Weissenberg camera, for which the layer line screen slot is formed by the displacement between the ends of two coaxial identical radii cylinders, the slot width is effectively defined by the inner edge of one cylinder and the outer edge of the other [Fig. 3(*a*)]. Thus the best value of R is the mean of the internal and external radii of the cylinders. On the other hand, for the Nonius integrating Weissenberg camera the layer line screen slot is formed by the movement of two cylinders one inside the other. In this case, if the camera is inclined in a clockwise sense (when viewed from above), the slot width is defined by the inner edge of the outer cylinder and the outer edge of the inner cylinder [Fig. 3(*b*)]. Thus the best value of R is the inner radius of the outer cylinder. On the other hand if this Weissenberg camera is inclined anti-clockwise, the slot width is defined by the outer edge of the outer cylinder and the inner edge of the inner cylinder [Fig. 3(*c*)].

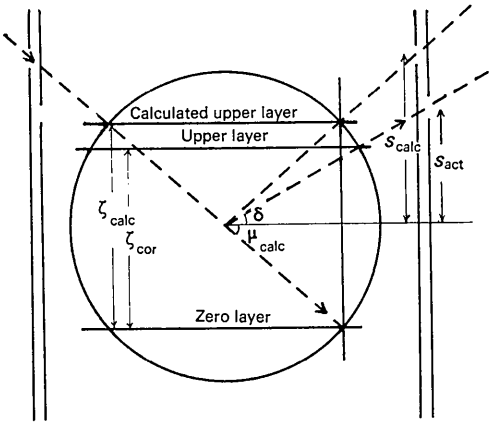

Fig. 2. Geometry of an equi-inclination Weissenberg camera in which an incorrect value of the equi-inclination angle has been used. The view is the same as before. The two positions of the layer line screen discussed have been slightly off-set laterally for clearness.

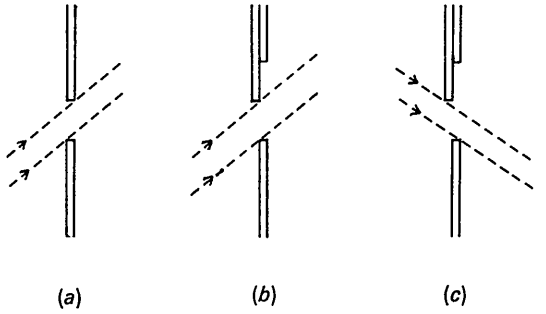

Fig. 3. Appearance of the diffracted beam and the layer line screen for (*a*) the Unicam Weissenberg camera, (*b*) the Nonius integrating Weissenberg camera inclined clockwise, (*c*) the Nonius integrating Weissenberg camera inclined anticlockwise.

Table 1 gives the results of applying the method twice, once in very favourable circumstances when a 0·2 mm diameter spherically ground crystal of struvite (initial ζ taken as 0·253) was examined on a Nonius integrating Weissenberg camera ($R = 23{\cdot}50$) and once in unfavourable circumstances when a 1 mm long needle-shaped crystal of roesslerite (initial ζ taken as 0·1325) was examined on a Unicam Weissenberg camera ($R = 23{\cdot}70$). In the latter case the needle was irradiated at its centre giving elongated spots.

Most of the terms in Table 1 are self-explanatory, except the last column. This contains the values of the equi-inclination angle obtained from accurately determined cell dimensions. These accurate cell dimensions were determined by Farquhar & Lipson's (1946) method from high order pinacoidal reflexions; the values obtained were plotted against the Nelson & Riley (1946) extrapolation function and extrapolated to a Bragg angle of 90°. The accuracy of the cell dimensions is estimated to be about 0·3%.

Comparison of the corrected and the accurately obtained values of the equi-inclination angle indicates that in a favourable case (*i.e.* struvite) the practical accuracy is high and agrees with the theoretical accuracy in spite of the need to interpolate when setting the equi-inclination angle, while in the case of roesslerite the practical accuracy is not as good as the theoretical one. This is, no doubt, due to the use of a needle-shaped crystal, as although the screen positions can be measured to the nearest 0·1 mm they cannot be set to this because of the elongation of the diffraction spots. In spite of the poorer accuracy, however, it can be seen that, in general, the corrected values of the equi-inclination

Table 1. *Comparison of corrected and accurately known values of the equi-inclination angle*

Substance	Layer	μ_{calc}	s_{calc} (mm)	s_{act} (mm)	δ	μ_{cor}	μ
Struvite	3	22°18′	9·6	9·5	22° 1′	22°10′	22° 8′
	4	30 24	13·8	13·5	29 52	30 8	30 9
	5	39 14	19·2	18·7	38 30	38 52	38 53
Roesslerite	5	19 21	8·3	8·3	19 18	19 19	19 32
	6	23 25	10·3	10·5	23 54	23 40	23 39
	7	27 38	12·3	12·8	28 22	28 0	27 54
	8	32 0	14·8	15·2	32 40	32 20	32 20
	9	36 36	17·6	18·3	37 40	37 8	36 59

angle are still considerably better than the initial ones. Presumably the accuracy could be increased further by irradiating only the tip of the crystal. Hence it would appear that this method is capable of high accuracy and, in addition, has two advantages over Sayre's (1954) method; namely, it is perfectly general and there is no need to take Weissenberg photographs before applying it. However, it has a disadvantage that incorrect settings have, in general, a larger effect on Sayre's parameter, $d\omega$, than the error in the layer line screen movement, leading, in theory, to lower accuracy. To some extent this is counteracted by the fact that the spots on the Weissenberg photograph are likely to be more elongated than those on the oscillation photograph, owing to strain (combined with the velocity factor) and to the Phillips (1954) effect.

In practice, of course, one need not take photographs of each layer line, only of the most accurate.

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The Crystal and Molecular Structure of Neothiobinupharidine Dihydrobromide Tetrahydrate *

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Crystals of neothiobinupharidine dihydrobromide tetrahydrate, $C_{30}H_{42}O_2N_2S.2HBr.4H_2O$, are orthorhombic, space group $P2_12_12_1$, with $a=14{\cdot}71$, $b=16{\cdot}07$, $c=14{\cdot}68$ Å. The structure was solved by the heavy-atom method and the refinement was carried out by the least-squares procedure. The final R value is 0·097. All six-membered rings are in chair conformation and all substituents in equatorial positions. There is some molecular disorder associated with an approximate non-crystallographic twofold axis. The alkaloid molecules are embedded between hydrogen-bonded sheets composed of water molecules and bromide ions.

Introduction

A new class of natural products was discovered by Achmatowicz & Bellen (1962), who isolated four alkaloids containing sulphur from *Nuphar luteum* (L) *Sm.* (yellow water lily). Subsequently, Achmatowicz & Wróbel (1964) isolated another substance from the same source which with empirical formula $C_{30}H_{42}O_2N_2S$ and this was named neothiobinupharidine. The authors investigated this compound using ultraviolet, infrared and nuclear magnetic resonance spectra, but it was mostly on the basis of mass spectra that Achmatowicz, Banaszek, Spiteller & Wróbel (1964) proposed a complete structure (I). At the same time, Prof. Achmatowicz very kindly sent us crystals of neothiobinupharidine dihydrobromide tetrahydrate for X-ray structure analysis. This investigation revealed a somewhat different structure (II) which was first reported in a preliminary communication (Birnbaum, 1965).

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