Short Communications

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Seminvariants for space groups $I\bar{4}2m$ and $I\bar{4}2d$.

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Errors in the tables of seminvariants given by Karle & Hauptman [*Acta Cryst.* (1961). **14**, 217–223] are corrected.

There are two errors in the tables of seminvariants given by Karle & Hauptman (1961) which seem to have continued unnoticed (*e.g.* by Giacovazzo, 1974 and in *International Tables for X-ray Crystallography*, 1974). The space group $I\bar{4}2m$, No. 121 (*International Tables for X-ray Crystallography*, 1969) has its origin fixed by convention at positions with point symmetry $\bar{4}2m$. There are only two such distinct equivalent points in the unit cell, $0,0,0$ and $0,0,\frac{1}{2}$. The space group $I\bar{4}2d$, No. 122 (*International Tables*, 1969) has its origin fixed by convention at positions with point symmetry $\bar{4}$. There are four such distinct points in the unit cell, but they are not all equivalent. The disposition of symmetry elements about the equivalent points $0,\frac{1}{2},\frac{1}{4}$ and $0,\frac{1}{2},\frac{3}{4}$ differs from that about the equivalent points $0,0,0$ and $0,0,\frac{1}{2}$. The latter pair of points are thus the only permissible origins for this space group.

(Note that when conventional body-centred unit cells are chosen the points $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ and $\frac{1}{2},\frac{1}{2},0$ are translationally equivalent to the points $0,0,0$ and $0,0,\frac{1}{2}$, and are not distinct origins.)

It is therefore clear that for both $I\bar{4}2m$ and $I\bar{4}2d$ the seminvariant vector is (l), the seminvariant modulus is (2), and that these two space groups are not of type $3P_34$ but of type $3P_32$ in Karle & Hauptman's notation, $(l)-I(2)$ in Giacovazzo's notation.

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Absorption correction for back reflexions in plate-shaped crystals of high absorption.

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A simple expression for the absorption correction, derived for an infinite plate intercepting the whole beam, has been shown to be applicable in the structure analysis of plate-shaped hexagonal polytypes of high absorption coefficient. The expression holds almost exactly for crystals which have even a slightly bigger cross section than the X-ray beam, while for crystals completely bathed in X-rays the expression, with slight modification, holds approximately.

Some authors have described more or less general methods for calculating the absorption corrections (*e.g.* Howells, 1950), while others have calculated the correction for suitably shaped crystals, *e.g.* Fitzwater (1961) for ellipsoidal crystals. However, as a result of one or more difficulties in the application of these methods, such as the tediosity of the method, the impossibility of grinding the crystal to a particular shape, the size of the crystal being bigger than the cross section of X-ray beam, some authors have preferred to devise their own simple treatments suited to particular cases (*e.g.* Ferrari, Braibanti & Tiripicchio, 1961). We have arrived at a simple expression for the absorption correction for the special case of back reflexions from highly absorbing platy crystals of a hexagonal polytype, such as CdI_2, PbI_2 and $CdBr_2$. Up until now in the absence of any suitable expression, the practice has been to omit the correction, *e.g.* in the crystal structure determination of 60, 11 and 4 polytypes of CdI_2, PbI_2 and $CdBr_2$ respectively, by different workers (Trigunayat & Verma, 1975). The expression is equally applicable to the plate-shaped crystals of other substances which have sufficiently high absorption coefficients. The underlying analytical treatment will be illustrated by the example of cadmium iodide crystals, typically 50–500 μm in thickness and 0·5 to 5·0 mm across, with **c** perpendicular to the plate.

Reflexions $01.l$ for the upper values of l, *viz.* $l=2n$ to $4n$

for a polytype nH or nR [such that the reflected beam emerges from the same face as the incident beam enters with a minimum glancing angle of $\psi = 2\theta + \varphi - 90$ (Fig. 1) of 5° (Table 1)] are sufficient for a complete structure analysis of the polytype (Verma & Krishna, 1966). These reflexions are recorded by oscillating the crystal about the a axis which lies in the plane of the crystal plate.

Table 1. *Values of* φ, θ *and the absorption correction factor in case of* CdI_2 *polytype* $4H$ *for* $01.l$ *spots* ($l = 8$ *to* 16)

l	φ(°)	θ(°)	$\dfrac{1}{1-\cos\varphi\,.\,\sec(2\theta+\varphi)}$	$\dfrac{1}{\sec\varphi-\sec(2\theta+\varphi)}$
8	35·24	29·81	0·0941	0·0769
9	34·25	33·28	0·1851	0·1530
10	32·63	36·96	0·2527	0·2129
11	30·44	40·87	0·3047	0·2625
12	27·70	45·07	0·3453	0·3057
13	24·39	49·64	0·3784	0·3446
14	20·40	54·72	0·4060	0·3805
15	15·51	60·56	0·4300	0·4144
16	9·14	67·77	0·4525	0·4468

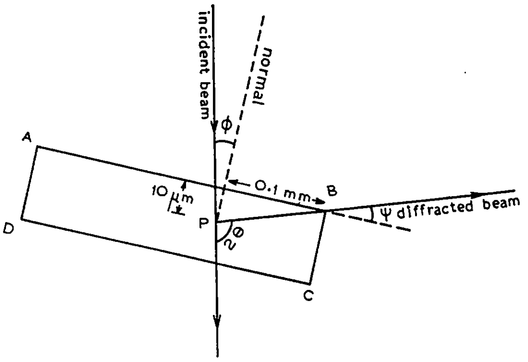

Fig. 1. Top section of the geometry of zero-layer back reflexion from a plate-shaped crystal mounted along an axis lying in the plane of the crystal plate.

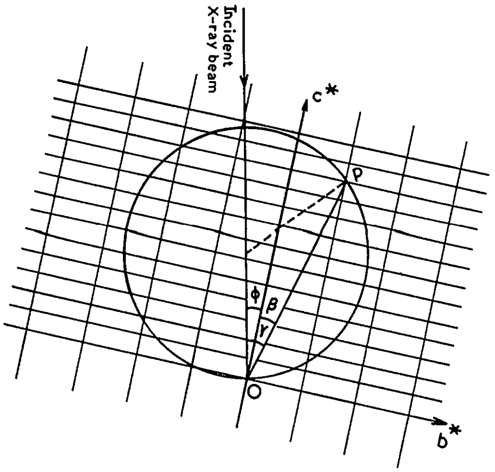

Fig. 2. Zero-layer b^*c^* reciprocal net and the sphere of reflexion, showing the geometry of origination of a $01.l$ reflexion from the lattice point P.

For Cu $K\alpha$ radiation, $\mu \simeq 1500$ and 95% of the reflected radiation therefore comes from a surface layer 10 μm thick. Less than 0·25% comes from layers more than 20 μm from the surface, even for normal incidence and reflexion. This means that for a large plate intercepting the incident beam, the emerging reflected beam will in the worst case only spread beyond the area of the incident beam by about 0·1 mm. A plate which is larger than the incident beam by this margin (allowing for obliquity, *i.e.* φ in Fig. 1 and Table 1) can therefore be treated as an infinite thick plate if it is 20 μm or more in thickness. The absorption correction factor for such a plate is (Jeffery, 1971).

$$A = \frac{1}{\mu[1-\cos\varphi\,.\,\sec(2\theta+\varphi)]}\,. \tag{1}$$

Even if the plate is smaller than the beam, the edge effects can be shown to be less than about 10–15% of the absorption correction for such plates. However, the area of the incident beam intercepted in this case is proportional to $\cos\varphi$ and the correction factor becomes,

$$A' = \frac{1}{\mu[\sec\varphi-\sec(2\theta+\varphi)]}\,. \tag{2}$$

Even in the case where it is doubtful whether the incident beam is completely intercepted, it is better to apply correction (1) than no correction at all, since the error involved will be much smaller. Typical calculations (derived from the Appendix) are shown in Table 1.

APPENDIX

The angle φ occurring in expressions (1) and (2) can be determined as follows. Fig. 2 shows the sphere of reflexion of radius unity and the zero-layer b^*c^* reciprocal net of the crystal. Let P be the point giving rise to a particular $01.l$ reflexion and O be the origin of the reciprocal lattice. From the geometry we have

$$OP = d^*_{01.l} = \sqrt{b^{*2} + l^2c^{*2}} = 2\sin\theta$$
$$\gamma = 90 - \theta$$
$$\beta = \tan^{-1} b^*/lc^*$$
$$\varphi = \gamma - \beta\,.$$

The value of A or A' is calculated from (1) or (2) and the values of θ and φ. The observed intensities are then divided by A to A' to give a corrected set of intensities for comparison with calculated values. For relative intensities, μ can be given any convenient value.

The expressions for A, A', θ and φ can be easily extended to non-zero layers.

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