RAPID DETERMINATION OF INTERPLANAR SPACINGS FOR TRIMETRIC CRYSTALS

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Abstract

A rapid and accurate technique of determining interplanar spacings for trimetric crystals by the use of a grid based on Peacock's graphical method is described. Interplanar spacings of chalcanthite were determined by such grid measurements and showed remarkable agreement with the calculated values. The procedure for keeping the method at its highest degree of accuracy is discussed. Miller indices are assigned to some of the lines of a powder film of chalcanthite on the basis of the spacing values.

Introduction

A graphical method of determining the interplanar spacings in trimetric lattices, given the values of the interaxial angles and the unit translations, has been described by M. A. Peacock (1938). This method is far more rapid than the laborious mathematical calculations necessitated by the standard formulae. Herewith is described a grid which greatly increases the speed of this graphical method yet maintains or improves its degree of accuracy. The ease with which lattices, regardless of symmetry, are handled by the method is illustrated by a triclinic example.

Principles of the Graphical Method

The procedure for the graphical method involves: (1) the drafting of a scale model of the reciprocal lattice, and (2) the measurement of distance between the lattice origin and the various "face poles" of the reciprocal lattice model.

The first step may be accomplished (Fig. 1a) by developing an ordinary gnomonogram into an orthographic projection of a gnomonic lattice[1] whose origin is at the center of the sphere of projection. On this scale the distance between adjacent levels of the reciprocal lattice (i.e. those parallel the plane of the gnomonogram) equals r, the radius of the fundamental sphere. These layers are termed 0-level, 1-level, 2-level, etc., according to the number of radii at which they are located above the origin. The 1-level in Fig. 1a is, of course, the gnomonogram of classical crystallography and the primed gnomonic projection constants p_0', q_0', etc., are used in its construction.

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[1] Here used to designate a reciprocal lattice on the same scale as a similarly oriented gnomonogram of the crystal.

The second step, measurement of the central distance[2] of the gnomonic lattice point, is accomplished essentially through use of cross sections similar to AB of Fig. 1b. These all involve right triangles (cf. OO_3P of Fig. 1b) with one apex at O, the lattice origin; another at O_n, the orthographic projection of O on the n-layer; and the third apex at P, a random lattice point in this n-layer which reciprocally represents the set of crystal
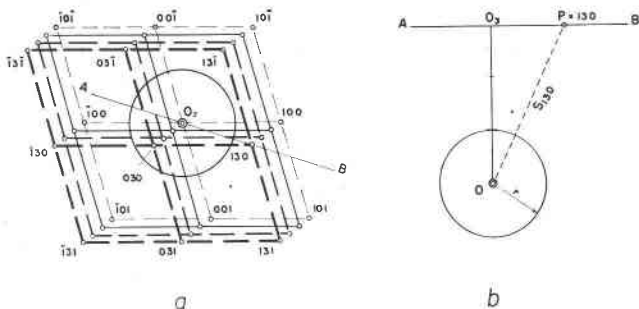

a b

Fig. 1. (a) Orthographic projection of a triclinic lattice upon the $|h3l|$ plane; thus [b] is normal to the plane of the paper. The equator of the fundamental sphere appears as the circle centered on O_3, which is the projected position of the lattice origin ($=$ center of sphere) upon the $3d$ level.

To add apparent depth to the lattice, the lines denoting the upper layers are successively heavier. Removal of all dashed lines and of line AB would leave a gnomonogram, with P' extending to right and R' extending downwards.

(b) A cross section through line AB of (a). O_3 is directly above O, the lattice origin, a distance of $3r$, where r is the radius of the fundamental sphere. For this [b]-vertical gnomonic lattice, S_{130} represents the central distance of the point, (130), generalized as P.

planes whose interplanar spacing is desired. The lengths of the two legs, O_nP and OO_n, of the right triangle are easily determined since O_nP is located in the plane of the orthographic projection and therefore directly measurable while OO_n is equal to nr. The hypotenuse OP, which represents S_{hkl}, the central distance of point P, is then secured from the two legs by Peacock's (1938, pp. 97–98) clever graphic method.

The grid scales (Fig. 2) offer a further short cut. For this same point P, located in the n-layer of the gnomonic lattice, it is only necessary to measure the leg O_nP with a photographic positive of the n-level grid scale; the resulting grid scale reading, G_{hkl}, is the value of the ratio S_{hkl}/r.

PREPARATION OF GNOMONOGRAM FOR USE WITH GRID

The basic gnomonogram from which the gnomonic lattice is to be developed should be drawn with a fundamental sphere of a radius equal to

[2] The distance of the point from the gnomonic lattice origin.

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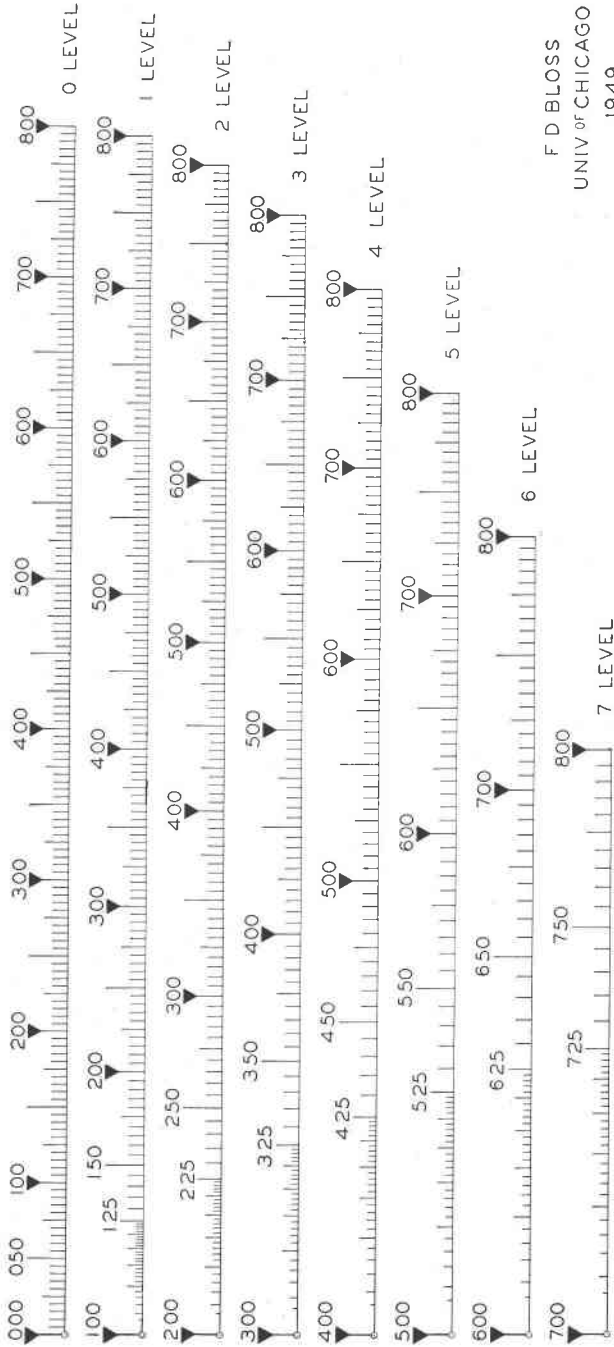
Fig. 2. The grid scales used for determining the G_{hkl} values, i.e., the ratio of S_{hkl}, the central distance, to r, the vertical distance between levels of the lattice.

unit distance on the 0-level grid scale. The vertical distance between layers of the gnomonic lattice, which always equals r, thus becomes equal to 1 unit on the 0-level grid scale, a condition necessary to permit use of the grid scale. This is most easily done by calculating the gnomonic projection elements (Table 1) for unit radius and then setting off these distances in terms of 0-level scale units of the positive copy (Fig. 2) being used for the measurements.

That gnomonogram which has the direct axis of largest unit translation normal to it will be found to yield the most accurate results; reasons for this are discussed in a later section.

The development of a gnomonogram into an orthographic projection of a gnomonic lattice is readily apparent from Fig. 1a. A more quickly prepared substitute (Fig. 3) of equal utility may be developed merely by assuming the basic gnomonogram to represent different levels $(0, 1, 2 \cdots n)$ of the gnomonic lattice and locating the orthographically projected positions $(O, O_1, O_2, \cdots O_n)$ of the lattice origin upon each of these layers. These projected positions are quickly located. For example, the origin of the 0-level coincides with the projected and actual positions of the gnomonic lattice origin; for the remaining levels, projected lattice origin positions $(O_1, O_2, \cdots O_n)$ are located along a line extending outward from the origin of the basic gnomonogram at a ϕ angle equal but opposite in sign to that of the pinacoid nearest horizontality; they are periodically spaced outward along it, in order of their subscripts, at intervals equal to tan ρ in 0-level units for this cited pinacoid.

Because of this convention of considering the gnomonogram to represent different layers, it is convenient to label its points with only partial Miller indices, leaving a blank for the index referring to the crystal axis normal to the gnomonogram.

PREPARATION OF GRID

Prepare a positive of Fig. 2 on heavy base film, preferably so it can be used with the emulsion side down (to reduce parallax).[3] A thin coat of shellac sprayed on the emulsion side will protect it from wear and moisture. Carefully pierce the "minimum point" (encircled left end) of each grid scale with a fine sharp-pointed needle; use a magnifying lens to locate this hole exactly on the point.

MEASUREMENT OF G_{hkl} WITH THE GRID SCALES

G_{hkl} values for all the points in a given layer, e.g., the n-level, can be secured rapidly by transfixing the "minimum point" of the n-level grid

[3] Copies from a glass plate negative of Fig. 1 are available from the author at the cost of preparation and handling.

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scale (Fig. 2) to the O_n point of the orthographically projected gnomonic lattice and then slowly rotating this scale around O_n. On a form similar to that used in Table 2, record the Miller indices and the values of the intercepts of the points on the n-scale during its rotation. The former are

TABLE 1

Axis normal to gnomonogram	Elements to be calculated in order to construct gnomonogram		Orientation of reciprocal axes in gnomonogram		
	Symbol	Formula number[a]	Symbol	Coordinates	
				Phi	Rho
Standard Orientation					
c	γ^*	(3)	a^*	$+\gamma^*$	90°
	p_0'	(10)	b^*	0°	90°
	q_0'	(9)			
	$\tan \rho_0$	(4)			
	ϕ_0	(5)			
First Permutation					
a	α^*	(1)	b^*	$+\alpha^*$	90°
	q_1'	(17)	c^*	0°	90°
	r_1'	(18)			
	$\tan \rho_1$	(12)			
	ϕ_1	(13)			
Second Permutation					
b	β^*	(2)	a^*	0°	90°
	p_2'	(26)	c^*	$+\beta^*$	90°
	r_2'	(27)			
	$\tan \rho_2$	(21)			
	ϕ_2	(22)			

[a] Refer to formulae in Fisher (1952) from which element may be calculated.

secured by inserting the value of n into the blank of the partial Miller index. To measure G_{hkl} values for points in the n-1 layer, transfix the minimum point of the n-1 level scale to the O_{n-1} point and repeat the above procedure. Figure 3 shows the grid in a position to read G_{h4l} values for the points in a gnomonic lattice normal to [b].
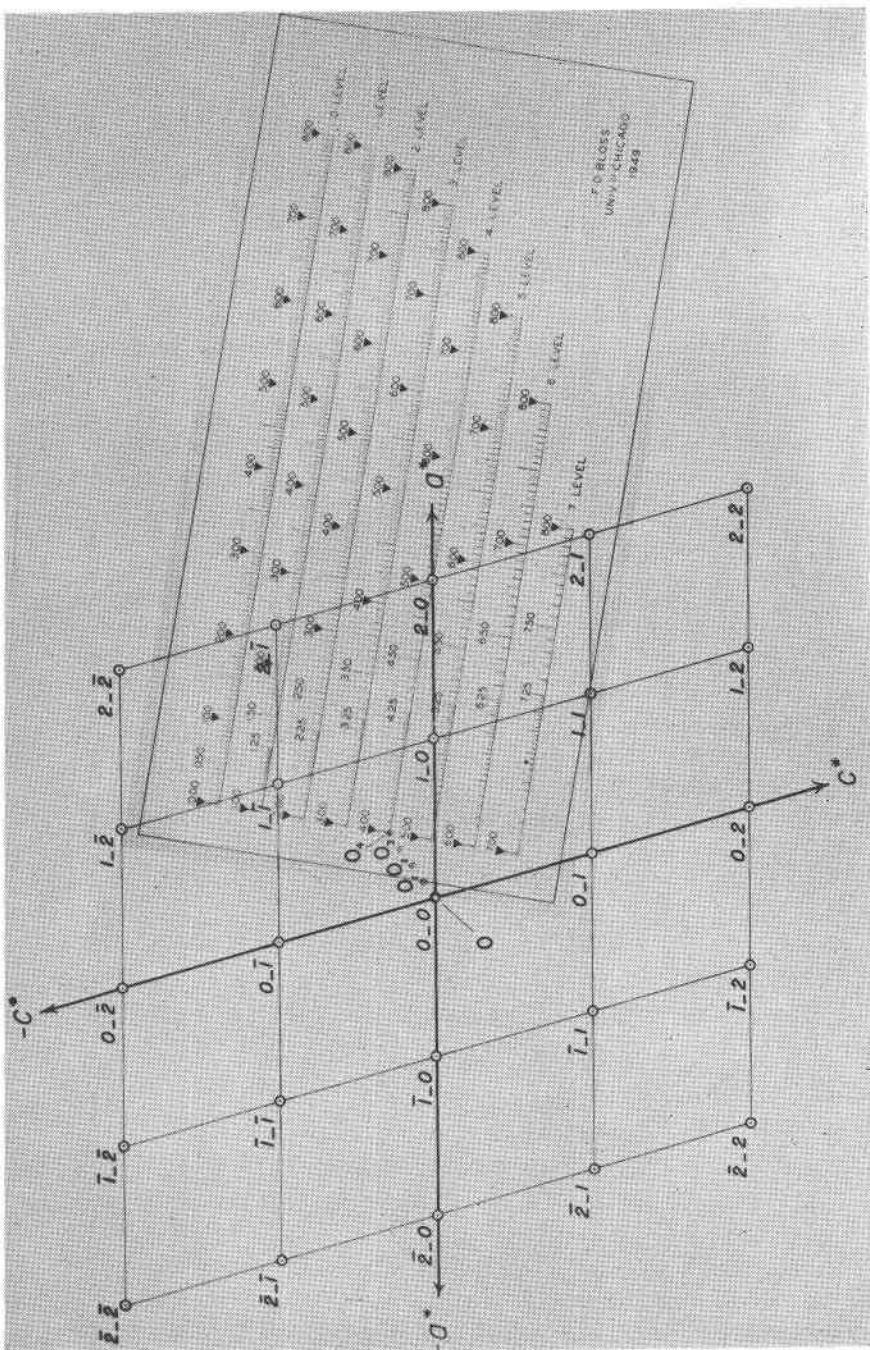
Fig. 3. Conversion of ordinary gnomonogram (center of its sphere of projection would be at O_1 and blanks in indices would be filled with 1's) into the equivalent of a series of orthographic projections of the lattice origin upon various levels of the reciprocal lattice. Thus O_1, O_2, O_3, represent these projected origin positions as the gnomonogram is respectively considered to represent the 1st, 2nd, and 3rd layers of the gnomonic lattice.

The grid has been placed over it with the 4-level scale in position to measure G_{hkl} values for all the "face poles" in the gnomonic lattice plane $|hkl|$ merely by rotation around the projected origin position, O_4. The pin ordinarily used to transfix the minimum point of the scale to O_4 has here been removed for illustrative purposes only. G_{230} can be seen to be equal to 5.02.

RELATIONSHIP BETWEEN G_{hkl} AND d_{hkl}

For the unit reciprocal lattice where the proportionality constant is set equal to unity (Buerger, 1942, p. 117), the following relations hold:

$$d_{001}{}^* = \frac{1}{c_0}, \quad d_{100}{}^* = \frac{1}{a_0}, \quad \text{and} \quad d_{010}{}^* = \frac{1}{b_0}.$$

For the various gnomonic lattices $d_{001}{}^G = r$, $d_{100}{}^G = r$, or $d_{010}{}^G = r$ depending upon whether the c, a, or b axis is oriented normal to the basic gnomonogram. Thus for these three different orientations, the derived gnomonic lattices represent vastly increased models of the unit reciprocal lattice on a scale $rc_0 : 1$, $ra_0 : 1$, or $rb_0 : 1$ respectively; or in general $ru_0 : 1$, where u_0 is the unit translation along the direct crystal axis normal to the gnomonogram.

Thus to translate S_{hkl}, a central distance measured in the gnomonic lattice, into σ_{hkl}, the analogous distance in the unit reciprocal lattice, it is necessary to divide the former by ru_0 which is the magnification constant, i.e., $\sigma_{hkl} = S_{hkl}/ru_0$. Therefore, since $\sigma_{hkl} = S_{hkl}/r$ and since by definition $d_{(hkl)} = 1/\sigma_{hkl}$, $d_{(hkl)} = u_0/G_{hkl}$. A 20-inch slide rule is generally adequate for this calculation. For the example of Fig. 3,

$$d_{(240)} = \frac{b_0}{G_{240}} = \frac{10.72A}{5.02} = 2.14 \text{ Å}.$$

The magnification constant, ru_0, of a gnomonic lattice is largest, and therefore the accuracy in this graphical determination of $d_{(hkl)}$ values is highest, when the gnomonic lattice is developed from a gnomonogram which is normal to the direct crystal axis of largest unit translation. Thus the basic gnomonogram for trimetric crystals with axes chosen as recommended by Donnay (1943) should generally be one normal to [b]. The ratios b_0/a_0 and b_0/c_0 indicate the ratio of size of a [b] vertical gnomonic lattice to those lattices developed from [a] and [c] vertical gnomonograms respectively. They thus indicate the relative importance of a constant plotting or measuring error in an [a] and a [c] vertical gnomonic lattice with respect to the same error in the [b] vertical one.

BRAVAIS SPHERE

Most of the planes encountered in morphology are those of maximum spacing and therefore with simple Miller indices. They are represented in the reciprocal lattice by the points closest to the origin. To avoid needless measurement of closely spaced planes it is well to decide upon a minimum spacing, d_{min}, and upon a device to avoid measurement of planes spaced less than this minimum value.

The grid is automatically such a device, supplanting the limiting circles

Peacock constructed for this purpose. A minimum spacing limitation of direct lattice planes is expressed in the reciprocal lattice by a limiting maximum distance from the origin, beyond which no points will be measured. Thus the choice of a value for d_{min} automatically circumscribes in the gnomonic lattice an origin-centered sphere (Bravais sphere of Peacock, 1938, pp. 95–96) within which all points will have scale readings less than G_{max}, where $G_{max} = u_0/d_{min}$ in grid units. Only the points in the gnomonic lattice denoting planes having spacings greater than d_{min} will have $G_{hkl} < G_{max}$ and therefore fall within the sphere.

EXAMPLE

Three differently oriented [a], [b], and [c] normal gnomonograms were constructed from Fisher's (1952) data on chalcanthite. The one normal to [b] is reproduced in Fig. 3 with the partial Miller indices indicated. The minimum spacing decided upon was 2.41 Å. Since the size of the gnomonogram and therefore the gnomonic lattice varies with the orientation, the value of G_{max} must similarly vary. For the gnomonic lattice with [a] normal,

$$G_{max} = \frac{a_0}{d_{min}} = \frac{6.104 \text{ Å}}{2.41 \text{ Å}} = 2.53;$$

for the gnomonic lattice with [b] normal,

$$G_{max} = \frac{b_0}{d_{min}} = \frac{10.72}{2.41} = 4.45;$$

and for the gnomonic lattice with [c] normal

$$G_{max} = \frac{c_0}{d_{min}} = \frac{5.949}{2.41} = 2.47.$$

Thus, for Fig. 3 no points, excluding the example (240), were measured if their G_{hkl} values exceeded 4.45; for the [c] normal gnomonic lattice, no points were measured if their G_{hkl} values exceeded 2.47, etc.

Table 2 presents the results of grid scale determinations on two differently oriented gnomonic lattices of chalcanthite. Results for the [a] normal gnomonic lattice were similar in accuracy to those for the [c] normal gnomonic lattice and therefore omitted. As was expected, results for the [b] normal gnomonic lattice are more accurate in general. The column entitled "d_{calc}" refers to the interplanar spacings calculated from the formula

$$\frac{1}{d_{hkl}{}^2} = h^2 a^{*2} + k^2 b^{*2} + l^2 c^{*2} + 2\,k\,l\,b^*c^* \cos \alpha^* + 2\,l\,h\,c^*a^* \cos \beta^* + 2\,h\,k\,a^*b^* \cos \gamma^*$$

(Int. Tables, 1935, p. 69) using Fisher's (1952) data.

TABLE 2

Miller indices	c-axis normal gnomonic lattice		b-axis normal gnomonic lattice		d_{calc} (from formula)
	G_{hkl}	$\dfrac{c_0}{G_{hkl}}$	G_{hkl}	$\dfrac{b_0}{G_{hkl}}$	
010	0.57	10.44	1.027	10.44	10.44
100	1.035	5.75	1.87	5.73	5.73
001	1.05	5.67	1.89	5.67	5.67
110	1.08	5.51	1.95	5.50	5.48
020	1.14	5.22	2.055	5.22	5.22
0$\bar{1}$1	1.15	5.18	2.08	5.15	5.14
011	1.235	4.82	2.205	4.86	4.84
$\bar{1}$01	1.26	4.72	2.26	4.75	4.74
$\bar{1}\bar{1}$1	1.25	4.76	2.27	4.72	4.72
$\bar{1}$10	1.27	4.69	2.30	4.66	4.67
120	1.38	4.31	2.505	4.28	4.28
$\bar{1}$11	1.49	4.00	2.675	4.01	4.00
02$\bar{1}$	1.48	4.02	2.69	3.99	3.98
$\bar{1}$21	1.49	4.00	2.70	3.97	3.96
021	1.61	3.69	2.88	3.72	3.71
101	1.67	3.56	3.00	3.57	3.56
1$\bar{2}$0	1.67	3.56	3.025	3.54	3.54
030	1.71	3.48	3.08	3.48	3.48
111	1.73	3.44	3.10	3.46	3.45
1$\bar{1}$1	1.79	3.33	3.25	3.30	3.30
130	1.82	3.27	3.29	3.26	3.26
$\bar{1}$21	1.88	3.17	3.37	3.18	3.18
$\bar{1}$31	1.87	3.18	3.405	3.15	3.15
0$\bar{3}$1	1.925	3.09	3.495	3.07	3.07
121	1.95	3.05	3.51	3.05	3.05
$\bar{2}\bar{1}$1	1.99	2.99	3.61	2.97	2.98
210	2.05	2.90	3.69	2.91	2.91
$\bar{1}\bar{1}$2	2.05	2.90	3.695	2.90	2.90
$\bar{2}$01	2.05	2.90	3.69	2.91	2.90
$\bar{1}$02	2.073	2.87	3.723	2.88	2.88
200	2.075	2.87	3.74	2.87	2.87
031	2.08	2.86	3.725	2.88	2.87
1$\bar{2}$1	2.07	2.87	3.77	2.84	2.85
002	2.10	2.83	3.775	2.84	2.83
$\bar{2}\bar{2}$1	2.095	2.84	3.80	2.82	2.82
0$\bar{1}$2	2.13	2.79	3.85	2.79	2.79
1$\bar{3}$0	2.16	2.76	3.89	2.76	2.75
220	2.16	2.76	3.92	2.74	2.74
$\bar{1}\bar{2}$2	2.18	2.73	3.945	2.74	2.72
012	2.22	2.68	3.98	2.69	2.69

TABLE 2—*Continued*

Miller indices	c-axis normal gnomonic lattice		b-axis normal gnomonic lattice		d_calc (from formula)
	G_{hkl}	$\dfrac{c_0}{G_{hkl}}$	G_{hkl}	$\dfrac{b_0}{G_{hkl}}$	
$\bar{1}12$	2.246	2.65	4.02	2.67	2.66
$\bar{2}11$	2.25	2.64	4.055	2.65	2.65
$2\bar{1}0$	2.25	2.64	4.07	2.64	2.64
040	2.28	2.61	4.107	2.61	2.61
131	2.31	2.58	4.14	2.59	2.58
$0\bar{2}2$	2.31	2.58	4.18	2.56	2.57
140	2.325	2.56	4.18	2.56	2.56
$\bar{1}31$	2.35	2.53	4.19	2.56	2.55
$\bar{1}41$	2.335	2.55	4.24	2.53	2.53
$\bar{2}31$	2.34	2.54	4.25	2.52	2.53
230	2.43	2.45	4.38	2.45	2.45
$0\bar{4}1$	2.43	2.45	4.40	2.44	2.44
$\bar{1}32$	2.44	2.44	4.42	2.43	2.43
$2\bar{1}2$	2.45	2.43	4.435	2.42	2.43
022	2.47	2.41	4.425	2.42	2.42
$1\bar{3}1$	2.46	2.42	4.45	2.41	2.41

ASSIGNMENT OF MILLER INDICES TO POWDER FILM LINES

Several powder films (114 mm. diameter camera) of chalcanthite were measured and the d values of the more intense lines are given in Table 3. Lines with d values of less than 2.41 Å were not measured. The graphically determined d values of Table 2 were then used to determine, where possible, the Miller indices of the plane (or planes) responsible for the respective powder lines.

Comparison of Tables 2 and 3 indicates that one may often unequivocally assign a set of Miller indices to the powder lines of greater d values whereas this possibility lessens as the powder lines of smaller d values are considered. However, extinction data may help eliminate some of the ambiguity. The powder line of $d = 2.55$ Å, e.g., could be caused by any one or combination of the planes $(0\bar{2}2)$, (140), $(\bar{1}31)$, $(\bar{1}41)$, and/or $(\bar{2}31)$. The spread of the several calculated or graphically measured d values around 2.55 Å is such that the disparity between any of them and the 2.55 Å powder photo measured value could be caused by small inaccuracies in the single crystal data, in the measurement of the powder line, or in the graphical determination of the d value. This latter effect may be eliminated by calculating the ambiguous d value from the stand-

TABLE 3

d measured from powder films	I/I_1 by visual estimation of intensities	Assignment of Miller indices to powder lines on basis of Table 2
10.45 Å	.3	010
5.70	.6	100 and/or 001
5.45	.7	110
5.15	.4	0$\bar{1}$1
4.70	1.0	$\bar{1}\bar{1}$1 (less likely 1$\bar{1}$0)[a]
4.26	.4	120
3.97	.7	0$\bar{2}$1 and/or $\bar{1}$21
3.69	.7	021
3.53	.35	1$\bar{2}$0[b] and/or 101
3.45	.3	111[a]
3.29	.4	1$\bar{1}$1[a]
3.24	.3	130
3.17	.2	$\bar{1}$21 and/or $\bar{1}$31
3.04	.4	121 and/or 0$\bar{3}$1
2.89	.05	210, $\bar{1}\bar{1}$2 and/or $\bar{2}$01
2.86	.15	200;[b] possibly (031, $\bar{1}$02 and/or 1$\bar{2}$1)
2.82	.4	$\bar{2}\bar{2}$1 and/or 002
2.78	.15	0$\bar{1}$2
2.74	.4	220 and/or 1$\bar{3}$0
2.72	.05	$\bar{1}$22
2.66	.4	2$\bar{1}$0[b] possibly ($\bar{2}$11 and/or $\bar{1}$12) also 012
2.55	.15	Numerous possibilities
2.52	.05	Numerous possibilities
2.45	.3	Numerous possibilities
2.41	.6	Numerous possibilities

[a] Crystal with $[c]$ parallel to the rotation axis was mounted in powder camera after removal of powder rod so that, in effect, a c-axis rotation picture was superimposed upon the powder film. No $(hk0)$ spot was observed to fall on this line.

[b] Under same conditions as above, an $(hk0)$ spot was observed to fall on or very near a powder line.

ard interplanar spacing formulae. Extinction data, if available, may be of considerable use in eliminating some of the ambiguities due to the cited inaccuracies.

ACKNOWLEDGMENTS

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