

# THE UNIT CELL AND SPACE GROUP OF PROBERTITE

WILLIAM H. BARNES,

*National Research Council, Ottawa, Ontario, Canada.*

## ABSTRACT

The unit cell constants and the space group of probertite,  $\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 5\text{B}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$ , have been determined by the Buerger precession method with the following results:  $a = 13.88 \text{ \AA}$ ,  $b = 12.56 \text{ \AA}$ ,  $c = 6.609 \text{ \AA}$ ,  $\beta = 107^\circ 40'$ ;  $a:b:c = 1.1053:1:0.5263$ ;  $Z = 2$ ; calculated density = 2.126 g./cc.; space group  $P2_1/n$ .

## INTRODUCTION

Probertite,  $\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 5\text{B}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$ , is monoclinic, has a radiating prismatic habit, perfect (110) cleavage and specific gravity of 2.141 (1). Axial ratios have not been determined, due presumably to the habit which makes the discovery of well-formed single crystals problematical. I am indebted to Professor Clifford Frondel for a specimen (No. 94710) from the Mineralogical Collection of Harvard University. The source locality was Lang, California.

## X-RAY DATA AND RESULTS

Assuming the cleavage plane to have been identified correctly from the point of view of the structural cell as (110), zero, first and second reciprocal lattice level photographs normal to the  $a$  and  $b$  axes were obtained with Professor M. J. Buerger's precession camera (2). Copper radiation, with a nickel foil filter, was employed. The films were measured with the appropriate measuring device (3).

The zero and first level photographs are reproduced in Figs. 1, 2, 3, 4 and it will be seen at once that the small specimen examined was a multiple growth consisting of several individuals rather than a single crystal. Some leakage of beta radiation through the foil also is apparent. The films illustrate an important application of the precession method based on the fact that there is no distortion of the plane nets in the photographs of the reciprocal lattice levels. Diffraction spots on a zero level photograph arising from a single crystal can be recognized without difficulty even in the presence of spots due to other individuals that may be present. The latter can be ignored thereafter and precise orientation of the selected crystal can be completed with the aid of subsequent precession photographs (2, p. 21 *et seq.*).

Since only a limited number of unit cell and space group studies have been made with the precession instrument the experimental data obtained in the present instance are reproduced in full. They illustrate typical

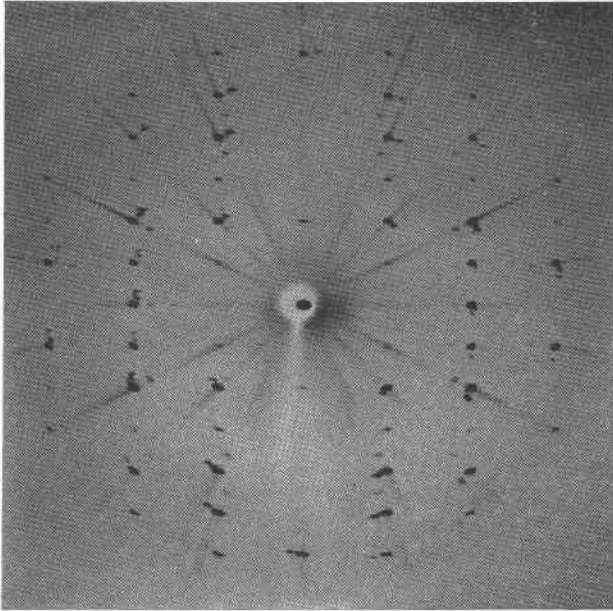


FIG. 1. Probertite.  $a$  axis, zero level (Cu) ( $c^*$  horizontal).

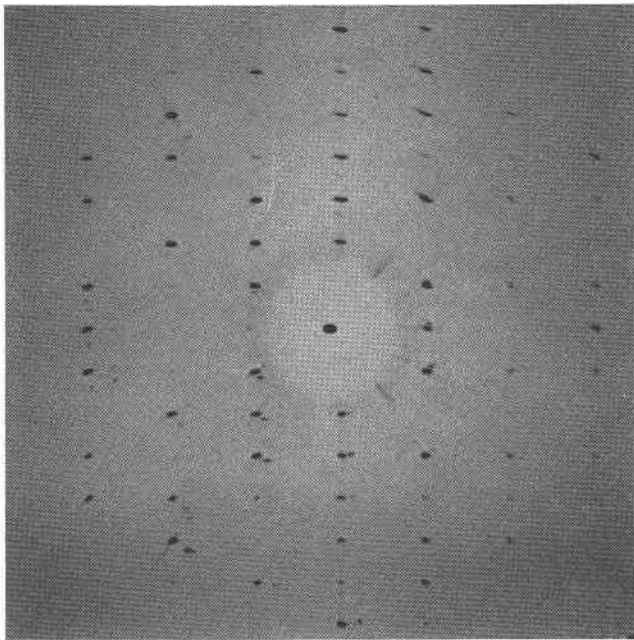


FIG. 2. Probertite.  $a$  axis, first level (Cu) ( $c^*$  horizontal).

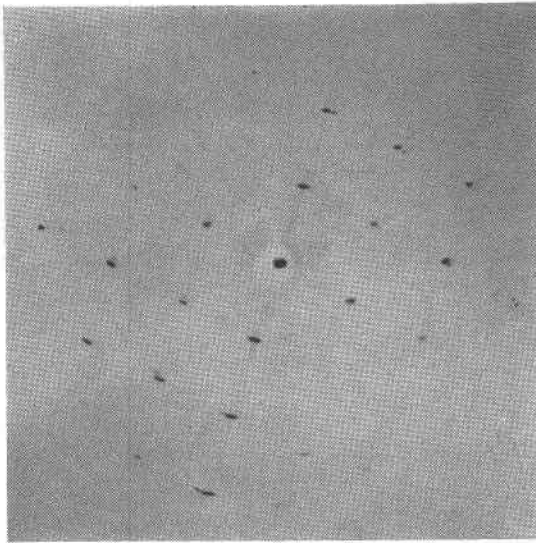


FIG. 3. Probertite.  $b$  axis, zero level (Cu) ( $c^*$  horizontal).

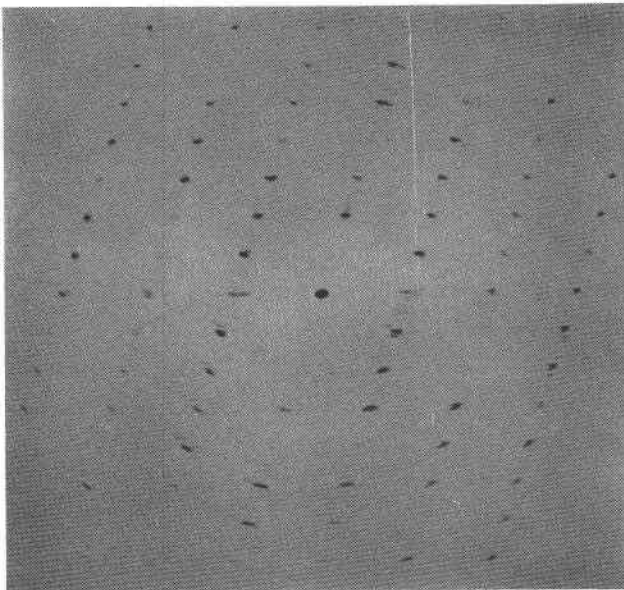


FIG. 4. Probertite.  $b$  axis, first level, (Cu) ( $c^*$  horizontal).

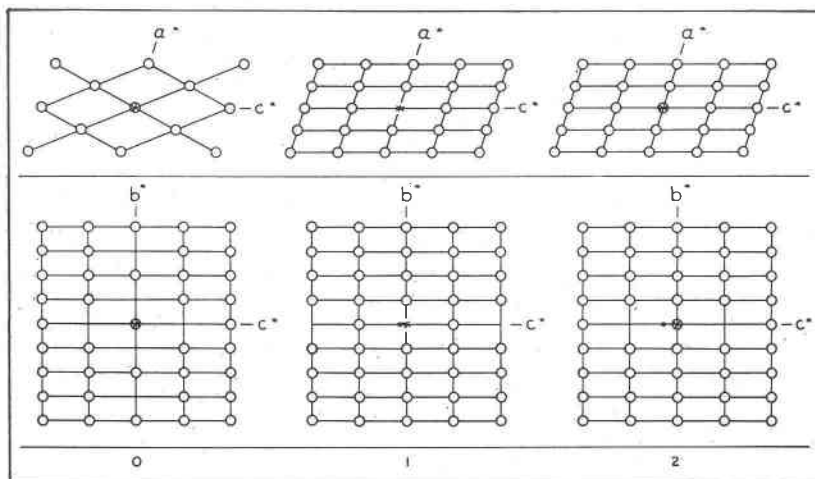


FIG. 5. 0,1,2 levels reciprocal lattice nets shown by (upper row)  $b$  and (lower row)  $a$  axis precession photographs of probertite. (Diffraction symbol  $(2/m)P(2_1/n)$ .)

measurements on average precession films and show that the use of a measuring device that reads (with verniers) to five minutes of arc and 0.05 mm. length is more than justified.

#### $a$ AXIS, ZERO LEVEL

*Angles between  $b^*$  and  $c^*$*

Scale reading	Angle
89°15'	—
359°00'	90°15'
268°50'	90°10'
178°55'	89°55'
89°15'	89°40'
<i>average</i>	90°00'

The net is orthogonal. Deviations from 90°00' can be ascribed to experimental error particularly since + and - deviations, respectively, are observed for adjacent (and not opposite) angles.

#### $c^*$ translation ( $l_c^*$ ) and spacing ( $d_c^*$ )

(Perpendicular distances between successive rows of spots; the second set of each  $Fl^*$  measurement was obtained after rotating the film through 180°)

Scale reading (cm.)	$Fl_c^*$ (cm.)	Scale reading (cm.)	$Fl_c^*$ (cm.)
12.030	—	11.980	—
10.560	1.470	10.510	1.470
9.080	1.480	9.040	1.470
7.620	1.460	7.580	1.460
6.145	1.475	6.105	1.475
<i>average</i>	1.471	<i>average</i>	1.469

average both sets = 1.470 cm.

$$Fd_c^* = Fl_c^* \sin \beta^*$$

$$= 1.470 \sin 72^\circ 20' = 1.4007 \text{ cm.}$$

*b*\* translation (*t*<sub>b</sub>\*) and spacing (*d*<sub>b</sub>\*)

Scale reading (cm.)	$Ft_b^* = Fd_b^*$ (cm.)	Scale reading (cm.)	$Ft_b^* = Fd_b^*$ (cm.)
12.600	—	12.885	—
11.860	0.740	12.160	0.725
11.125	0.735	11.430	0.730
10.385	0.740	10.690	0.740
9.650	0.735	9.970	0.720
8.900	0.750	9.220	0.750
8.175	0.725	8.480	0.740
7.435	0.740	7.750	0.730
6.695	0.740	7.010	0.740
5.965	0.730	6.265	0.745
5.230	0.735	5.525	0.740
<i>average</i>	0.737	<i>average</i>	0.736

average both sets,  $Fd_b^* = 0.7365$  cm.

$d_b^* = 0.7365/6.00 = 0.1228$  reciprocal lattice units.

*b* AXIS, ZERO LEVELAngles between *a*\* and *c*\*

Scale reading	$\beta^*$	$\beta$
17°40'	—	—
269°55'	—	107°45'
197°40'	72°15'	—
90°05'	—	107°35'
17°40'	72°25'	—
<i>average</i>	72°20'	107°40'

*c*\* translation (*t*<sub>c</sub>\*) and spacing (*d*<sub>c</sub>\*)

Scale reading (cm.)	$Ft_c^* = Fd_c^*$ (cm.)	Scale reading (cm.)	$Ft_c^* = Fd_c^*$ (cm.)
11.835	—	11.895	—
10.430	1.405	10.490	1.405
9.030	1.400	9.095	1.395
7.640	1.390	7.695	1.400
6.250	1.390	6.290	1.405
<i>average</i>	1.396	<i>average</i>	1.401

average both sets,  $Fd_c^* = 1.3985$  cm.

from *a* axis, zero level,  $Fd_c^* = 1.4007$  cm.

*average*,  $Fd_c^* = 1.3996$  cm.

$d_c^* = 1.3996/6.00 = 0.2333$  reciprocal lattice units.

$a^*$  translation ( $t_a^*$ ) and spacing ( $d_a^*$ )

Scale reading (cm.)	$Ft_a^* = Fd_a^*$ (cm.)	Scale reading (cm.)	$Ft_a^* = Fd_a^*$ (cm.)
13.115	—	12.340	—
12.440	0.675	11.670	0.670
11.780	0.660	11.015	0.655
11.115	0.665	10.350	0.665
10.440	0.675	9.680	0.670
9.780	0.660	9.015	0.665
9.105	0.675	8.350	0.665
8.445	0.660	7.685	0.665
7.770	0.675	7.015	0.670
7.115	0.655	6.345	0.670
6.450	0.665	5.685	0.660
5.780	0.670	5.010	0.675
average	0.666 <sub>9</sub>	average	0.666 <sub>4</sub>

average both sets,  $Fd_a^* = 0.6667$  cm.

$d_a^* = 0.6667/6.00 = 0.1111$  reciprocal lattice units.

Taking  $\lambda$  (CuK $\alpha$ ) as 1.5418 Å (4), the formula weight ( $M$ ) as 702.5 (2Na, 45.994; 2Ca, 80.16; 10B, 108.2; 20H, 20.160; 28O, 448.000) chemical atomic mass units and the density ( $\rho$ ) =  $MZ/1.6602V$  g./cc. (4) the direct cell constants are as follows:

$$a = \lambda/d_a^* = 1.5418/0.1111 = 13.88 \text{ \AA} \ddagger$$

$$b = \lambda/d_b^* = 1.5418/0.1228 = 12.56 \text{ \AA} \ddagger$$

$$c = \lambda/d_c^* = 1.5418/0.2333 = 6.609 \text{ \AA} \ddagger$$

$$\beta = 107^\circ 40'$$

$$a:b:c = 1.1053:1:0.5263$$

$$V \text{ (volume of unit cell)} = abc \sin \beta = 1097.2 \text{ \AA}^3$$

$$Z \text{ (formula units per cell)} = \rho V/1.6602M = \frac{2.141 \times 1097.2}{1.6602 \times 702.5} = 2.014 \approx 2.$$

calculated density = 2.126 g./cc.

‡ These values differ slightly from those reported at the 1947 Annapolis meeting of The Crystallographic Society of America (5) because they have been recomputed in terms of the currently adopted Å unit instead of the kX unit.

The central areas of the  $a^*c^*$  and  $b^*c^*$  plane nets for the zero, first and second reciprocal lattice levels normal to the  $b$  and  $a$  axes, respectively, are shown diagrammatically in Fig. 5, where extinctions other than those due to space group symmetry elements have been ignored as well as the relative intensities of diffraction spots. The center of each net is indicated by a star, which represents a point of intersection of the reciprocal lattice axis that is not contained within the net itself. The dot to the left of the center in the first and second level  $b^*c^*$  nets shows the point of intersection of the  $a$  (precession) axis, the increasing magnitude of the off-set

with increasing height of the level being due to the angle  $(90^\circ - \beta^*)$  between  $a$  and  $a^*$ ; in the other nets illustrated in Fig. 5 the point of intersection of the precession axis coincides with the center of the net.

It is at once apparent that the  $a^*c^*$  nets are characterized by a center of symmetry and the  $b^*c^*$  nets by two symmetry lines at right angles; the diffraction symmetry, therefore, is  $2/m$ . A comparison of the nets for the different levels shows that the direct cell as selected is a primitive one. The change from the diamond-like net for the  $b$  axis zero level to the rectangle-like net of the upper levels reveals an  $n$  glide plane of component  $a/2 + c/2$  perpendicular to the  $b$  axis. The doubled translation along  $b^*$  in the  $a$  axis zero level net indicates a  $2_1$  screw axis along  $b$ . The diffraction symbol, therefore, is  $(2/m)P(2_1/n)$  and the space group is  $P2_1/n (C_{2h}^5)$ .

#### ACKNOWLEDGMENTS

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