Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

Acta Cryst. (1959). **12**, 695

Unit cells and space groups for two etherates of sodium tridecahydrodecaborate(1-). By

HILLYER G. NORMENT, JR., *Callery Chemical Company, Callery, Pennsylvania, U.S.A.*

(*Received* 16 *April* 1959)

The discovery of the salt sodium tridecahydrodecaborate-(1-)($NaB_{10}H_{13}$) by Hough & Edwards (1958) has prompted further investigation of this compound with a view toward determination of its structure by single crystal X-ray methods. Attempts to prepare single crystals of the unsolvated material were unsuccessful due to its ease of hydrolysis and salt like properties. Stable macro-crystalline ethyl and methyl etherates have been prepared and preliminarily studied by single-crystal X-ray diffraction techniques.

Ethyl or methyl ether solutions of decaborane ($B_{10}H_{14}$) were reacted with slight excesses of sodium hydride at room temperature. After filtration to remove excess sodium hydride, excess ether was evaporated slowly to yield residues in which suitable single crystals were found.

By chemical analysis, the crystalline materials have been characterized as the mono-ethyl etherate,

$$NaB_{10}H_{13}.(C_2H_5)_2O\ ,$$

and the bis-methyl etherate,

$$NaB_{10}H_{13}.2\,(CH_3)_2O\ .$$

Both materials are extremely hygroscopic. The bis-methyl etherate must be stored in an undiluted atmosphere of methyl ether to prevent effervesence of methyl ether.

The mono-ethyl etherate crystals were found to be triclinic, with the following unit-cell parameters:

$$a = 5{\cdot}64 \pm 0{\cdot}02,\ b = 7{\cdot}42 \pm 0{\cdot}02,\ c = 9{\cdot}05 \pm 0{\cdot}03\ \text{Å};$$
$$\alpha = 83°\ 43' \pm 10',\ \beta = 86°\ 39' \pm 10',\ \gamma = 74°\ 0' \pm 10'\ .$$

Assuming one molecule per unit cell, the calculated density is 1·001 g.cm.$^{-3}$. The observed density is 0·983 g.cm.$^{-3}$. The space group is most certainly $P1$.

Crystals of the bis-methyl etherate were found to be monoclinic, space group $P2_1/c$, with unit-cell parameters:

$$a = 11{\cdot}49 \pm 0{\cdot}02,\ b = 29{\cdot}84 \pm 0{\cdot}02,\ c = 9{\cdot}72 \pm 0{\cdot}02\ \text{Å};$$
$$\beta = 94°\ 55' \pm 5'\ .$$

Space considerations indicate that there are eight molecules in the unit cell. The density calculated on this basis is 0·946 g.cm.$^{-3}$.

Weissenberg patterns of the bis-methyl etherate show an abnormal fall-off of reflection intensity with increasing Bragg angle. Very few reflections are observed beyond 90° of 2θ. This suggests high thermal motion and/or disorder, but a Debye–Scherrer pattern obtained at -100 °C. showed no indication of a phase transition.

Further work on these compounds is not anticipated in the near future.

Reference

HOUGH, W. V. & EDWARDS, L. J. (1958). Symposium on 'Borax to Boranes,' 133rd American Chemical Society Meeting, San Francisco.

Acta Cryst. (1959). **12**, 695

Crystal structures of the isostructural minerals lazulite, scorzalite and barbosalite.* By

MARIE L. LINDBERG and C. L. CHRIST, *U.S. Geological Survey, Washington 25, D.C., U.S.A.*

(*Received* 3 *April* 1959 *and in revised form* 23 *April* 1959)

Lazulite, $MgAl_2(PO_4)_2(OH)_2$, and scorzalite,

$$FeAl_2(PO_4)_2(OH)_2,$$

form a complete isomorphous series (Pecora & Fahey, 1949, 1950). Barbosalite, $FeFe_2(PO_4)_2(OH)_2$, the ferric analog of scorzalite (Lindberg & Pecora, 1954, 1955), is a third member of this isostructural series; no solid solution relations between barbosalite and the lazulite–scorzalite pair have been definitely established.

The present note describes the crystal structures found for the three minerals. Table 1 lists the chemical composition of the crystals used and the crystallographic data found in the current study. Visually estimated intensities obtained with Zr filtered Mo radiation were used for the structure determinations. A trial structure (common to the three minerals) was established by interpretation of the Patterson projections on the planes normal to *a*, *b*, and *c*, and from consideration of the known structure of the closely related tetragonal compound 'iron lazulite', $Fe_{3{\cdot}5}(PO_4)_2(OH)_2$, (Katz & Lipscomb, 1951). The trial structure was refined for lazulite and scorzalite by successive electron-density projections to yield the atomic parameters shown in Table 2. For

---

* Publication authorized by the Director, U.S. Geological Survey.

Table 1. *Data for lazulite, scorzalite, and barbosalite*

All monoclinic: $P2_1/c$–C^5_{2h} (No. 14)

	Lazulite	Scorzalite	Barbosalite
a	7·16[a] Å	7·15 Å	7·25 Å
b	7·26	7·31	7·46
c	7·24	7·25	7·49
β	120° 40′[b]	120° 35′	120° 15′
Cell contents	$Fe_{0\cdot23}Mg_{1\cdot77}Al_4(PO_4)_4(OH)_4$[c]	$Fe_{1\cdot53}Mg_{0\cdot47}Al_4(PO_4)_4(OH)_4$[c]	$Fe_2Fe_4(PO_4)_4(OH)_4$[d]
Density (calc.)	3·14 g.cm.$^{-3}$	3·32 g.cm.$^{-3}$	3·71 g.cm.$^{-3}$
Density (obs.)	3·12[c]	3·33[c]	3·60[d]
Source of crystal	Minas Gerais, Brazil	Victory, South Dakota	Minas Gerais, Brazil

[a] Lengths, ±0·3%, in Å.
[b] Angles, ±05′.
[c] J. J. Fahey, analyst (in Pecora & Fahey, 1950).
[d] M. L. Lindberg, analyst (in Lindberg & Pecora, 1954, 1955). (Observed density obtained on poor, fine-grained material).

Table 2. *Atomic parameters for lazulite and scorzalite*

Atom	x	y	z	
O_1	0·222	0·000	0·901	(lazulite)
	0·219	0·000	0·905	(scorzalite)
O_2	0·286	0·000	0·589	
	0·291	0·000	0·589	
O_3	0·048	0·253	0·130	
	0·046	0·254	0·129	
O_4	0·443	0·244	0·377	
	0·432	0·251	0·366	
OH	0·754	0·135	0·749	
	0·746	0·130	0·747	
P	0·250	0·113	0·749	
	0·249	0·111	0·747	
Al	0·732	0·266	0·004	
	0·732	0·267	0·004	
Mg or Fe	0	0	0	

$R_{h0l} = 0{\cdot}17$	$B_{h0l} = 0{\cdot}4$	(lazulite)
$= 0{\cdot}18$	$= 0{\cdot}6$	(scorzalite)
$R_{0kl} = 0{\cdot}18$	$B_{0kl} = 0{\cdot}8$	
$= 0{\cdot}14$	$= 0{\cdot}7$	

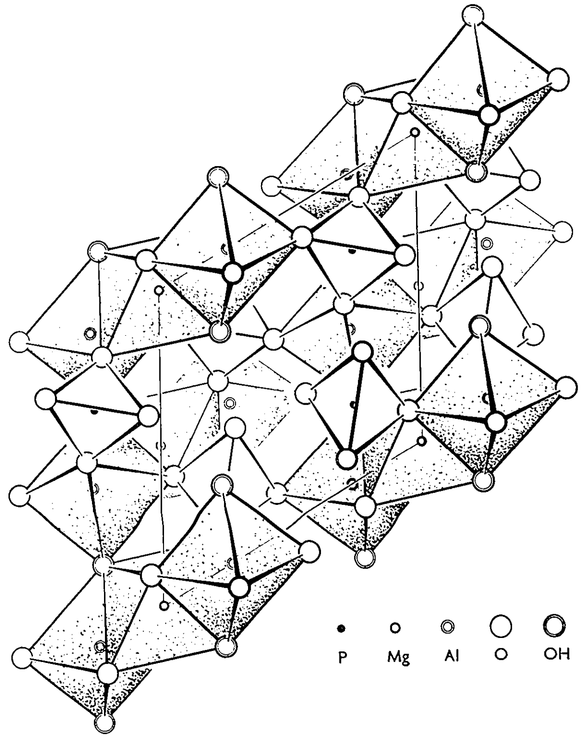

Fig. 1. Structure common to lazulite, scorzalite, and barbosalite, illustrated for lazulite, $MgAl_2(PO_4)_2(OH)_2$. The view is along [010]; a complete unit cell is outlined by the parallelogram, the origin being taken at the lower right-hand corner with z vertical and positive upwards, and x at 121° away from z and positive to the left. Certain of the polyhedra are omitted in order to reveal details of the structure. In lazulite, the average P–O length is $1{\cdot}5_5$ Å, the average Mg–O length is $2{\cdot}0_3$ A, and the average Al–O length is $1{\cdot}9_5$ Å.

barbosalite, only the electron-density projection normal to a has been studied so far. This projection is in excellent overall agreement with the corresponding ones for lazulite and scorzalite and yields approximately the same atomic parameters. At the present stage of refinement the residual factor $R_{0kl} = 0{\cdot}15$ and the average isotropic temperature factor $B_{0kl} = 0{\cdot}8$ Å^2 for barbosalite. X-ray patterns of the other two principal zones of barbosalite are completely analogous to those of lazulite and scorzalite.

A drawing of the basic structure common to the three minerals, using lazulite as the example, is shown in Fig. 1. The structure consists of the packing together of metal ion–oxygen octahedra and phosphate tetrahedra. The phosphorus is bonded to four non-equivalent oxygens. The proton present is assigned to the fifth independent oxygen. A magnesium–oxygen octahedron and two aluminum–oxygen octahedra share faces to form triple groups. These triple groups are linked together through corner-shared hydroxyls and through corner-shared oxygens of the phosphate groups. Each corner of a tetrahedral phosphate group is shared with a corner of an octahedral group in such a way as to tie the octahedral groups together laterally into sheets, and the sheets together into a three-dimensional edifice.

We are very grateful to two of our colleagues for much assistance: D. E. Appleman carried out many calculations on a digital computer, and H. T. Evans, Jr. prepared the drawing used in Fig. 1.

References

KATZ, L. & LIPSCOMB, W. N. (1951). *Acta Cryst.* **4**, 345.

LINDBERG, M. L. & PECORA, W. T. (1954). *Science*, **119**, 739.

LINDBERG, M. L. & PECORA, W. T. (1955). *Amer. Min.* **40**, 952.

PECORA, W. T. & FAHEY, J. J. (1949). *Amer. Min.* **34**, 83.

PECORA, W. T. & FAHEY, J. J. (1950). *Amer. Min.* **35**, 1.

Acta Cryst. (1959). **12**, 697

On the crystal structure of protactinium metal.

By JERRY DONOHUE, *Department of Chemistry, University of Southern California, Los Angeles 7, California, U.S.A.*

(*Received* 21 *November* 1958 *and in revised form* 24 *February* 1959)

The structure of protactinium has been determined by Zachariasen (1952) from powder data. The crystallographic description of this structure is that it is tetragonal with $a_1 = 3{\cdot}925$, $a_3 = 3{\cdot}238$ Å, space group *I*4/*mmm*, two atoms per unit cell. In this structure each atom has eight neighbors at 3·212 and two at 3·238 Å. Zachariasen pointed out that if the axial ratio had an ideal value of $\sqrt{\frac{2}{3}} = 0{\cdot}817$ instead of the observed value of 0·825, each metal atom would have ten neighbors at exactly the same distance.

Table 1. *Diffraction data*

	Observed		Calculated					
					Pa			
			PaO$_2$	PaO	Tetragonal		Orthorhombic	
	I	sin² θ	sin² θ	sin² θ	*hkl*	sin² θ	*HKL*	sin² θ
1	*vw*	0·0599	0·0588					
2	*ms*	0·0731		0·0725				
3	*w*	0·0787	0·0784		110	0·0772	020 200	0·0757 0·0767
4	*vs*	0·0975		0·0966	101	0·0953	111	0·0957
5	*w*$^+$	0·1567	0·1568		200	0·1544	220	0·1525
6	*wm*	0·1945		0·1933				
7	*vvw*	0·2174	0·2156					
8	*vw*	0·2293			002	0·2268	002	0·2302
9	*s*	0·2511			211	0·2497	131 311	0·2471 0·2492
10	*ms*	0·2679		0·2658				
11	*vw*	0·2917		0·2899				
							040	0·3029
12	*ms*	0·3064			112 220	0·3040 0·3088	022 400, 202	0·3059 0·3070
13	*m*	0·3841		0·3866	202 310	0·3812 0·3860	240 420, 222	0·3796 0·3827
14	*w*$^-$	0·4051			301	0·4041	331	0·4006
15	*w*	0·4599		0·4590				
16	*w*$^+$	0·4841		0·4832				
17	*w*	0·536			222	0·536	042 402	0·533 0·537
18	*w*	0·550			103	0·549	151	0·550
19	*w*$^+$	0·557			321	0·559	511, 113	0·556
20	*w*$^-$	0·580		0·580				
21	*m*	0·613			312	0·613	440, 242 422	0·610 0·613
					400	0·618		
22	*w*	0·652		0·652				
							060	0·681
					330	0·695	600	0·691
23	*s*	0·704			213	0·703	351	0·703
24		0·712			411	0·713	133, 531 313	0·708 0·710
25	*w*$^-$	0·772		0·773	420	0·772	260 620	0·758 0·766
26	*wm*	0·842		0·846	402	0·844	442	0·840
27	*w*$^-$	0·860			303	0·858	333	0·861
28	*vw*	0·872		0·870				
29	*vw*$^-$	0·905			004	0·907	062	0·911
30	*w*	0·921			332	0·922	602, 004	0·921