

THE RELATIONSHIP BETWEEN SENAITE, MAGNETOPLUMBITE, AND DAVIDITE¹ROLAND C. ROUSE AND DONALD R. PEACOR, *The University of Michigan, Ann Arbor, Michigan, 48104.*

ABSTRACT

Senaite has space group $R\bar{3}$ or $R\bar{3}$ with $a=10.42 \text{ \AA}$ and $c=20.86 \text{ \AA}$. Analysis of the implication function $I_3(xy0)$ together with chemical analyses shows that it has the probable formula $\text{Pb}(\text{Ti, Fe, Mn, Mg})_{24}\text{O}_{38}$ with $Z=3$ for the hexagonal cell. The structure is based on oxygen closest-packing with, by analogy to the magnetoplumbite structure, Pb in the anion framework. Davidite, which has a unit cell of the same dimensions as that of senaite, has the probable primitive unit cell contents $(\text{Ti, Fe, . . .})_{21}\text{O}_{39}$.

INTRODUCTION

An investigation of the mineral senaite has been undertaken as a part of a general study of the nature of polytypism in complex oxides. Of particular interest are the relationships among senaite, the chemically similar species davidite, magnetoplumbite, plumbiferite, and the synthetic hexagonal ferrites.

Senaite was first described by Hussak and Prior (1898) as a black sub-metallic mineral occurring as grains and rough crystals in the diamond-bearing sands at Dattas, Curralinho, and other localities near Diamantina, Minas Gerais, Brazil. Chemical analysis indicated that it had the formula $(\text{Fe,Pb})\text{O} \cdot 2(\text{Ti,Mn})\text{O}_2$, suggesting an analogy to the ilmenite group. The crystal symmetry was found to be that of class $\bar{3}$, while interfacial angles are similar to those of mohsite, a variety of ilmenite. Twinning on $\{11\bar{2}0\}$, as determined from morphological data, was found to be common. The crystals had a tabular or acute rhombohedral habit, the latter being unusual for ilmenite.

Hussak and Reitinger (1903) published additional information on the morphology and composition of large rough crystals. Chemical analyses on fresh crystals indicated the formula to be $(\text{Fe,Mn,Pb,Mg})\text{O} \cdot \text{TiO}_2$ and senaite was therefore assumed to be isomorphous with ilmenite. A complete correspondence was found with the morphology of crichtonite, a supposed variety of ilmenite. Bannister, Claringbull, and Hey (1954) have described crichtonite as an independent rhombohedral species with cell parameters $a \sim 37 \text{ \AA}$ and $c \sim 21 \text{ \AA}$. Freise (1930) published a chemical analysis of senaite from a diamond-bearing conglomerate near Diamantina.

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Strunz (1966) has grouped together davidite, crichtonite, senaite, and mohsite. For senaite the formula $(\text{Fe}^{2+}, \text{Fe}^{3+}, \text{Mn}, \text{Pb})_2(\text{Ti}, \text{Fe}^{3+})_6\text{O}_{12}$ and an axial ratio $c/a = 1.94$ are given. The value of a for crichtonite is shown to be $10.3\sqrt{13} \sim 37 \text{ \AA}$, corresponding to the pseudo- a of twinned davidite (Pabst, priv. comm.).

In the course of our investigation of polytypic relations among some complex oxides, we noted a correspondence in chemistry between senaite and the related closet-packed hexagonal mineral magnetoplumbite. Since magnetoplumbite is not isostructural with ilmenite, and since the data suggesting that senaite is isostructural with ilmenite were inconclusive, we suspected that senaite should be an independent species unrelated to ilmenite. We therefore undertook an investigation of senaite in order to define its relations with respect to chemically related phases.

UNIT CELL AND SPACE GROUP

Fragments of a rough single crystal of senaite from Dattas (Royal Ontario Museum #M12525) were examined with the precession camera. C -axis and a' -axis ($a\sqrt{3}$) photographs exhibit diffraction symmetry $R\bar{3}$. Zero level a' -axis photographs show pseudo- mm symmetry and there is therefore a pseudo-space group $R3m$, $R32$, or $R\bar{3}m$. The dimensions of the unit cell indexed on hexagonal axes are $a = 10.42 \pm 0.05 \text{ \AA}$ and $c = 20.86 \pm 0.1 \text{ \AA}$. For the rhombohedral cell $a_r = 9.19 \text{ \AA}$ and $\alpha = 69^\circ 02'$. Similar results were obtained from a crystal from Santa Quitéria, Brazil (Swedish Museum of Natural History #511095).

ANION PACKING RELATIONS

The cell parameter a equals 5.88 \AA for the hexagonal, chemically related mineral magnetoplumbite. Magnetoplumbite ($\text{PbFe}_{12}\text{O}_{19}$) has a structure based on oxygen closest-packing with the value of a corresponding to twice the oxygen separation within a closest-packed layer. Since the value of a of senaite is nearly twice the value of a for magnetoplumbite, it was initially assumed that this parameter corresponded to four times the oxygen interatomic distance.

In the course of an investigation of the crystal structure relations of senaite, the implication function $I_3(xy0)$ was obtained from the Patterson function $P(uv0)$. This function for space groups $R\bar{3}$ and $R3$ has peaks directly corresponding to those of the electron density projection $\rho(x,y)$ (Buerger, 1959). Figure 1 is a representation of this function with all large peaks shown as circles with radii proportional to their heights, and a_1 and a_2 corresponding to the senaite cell edges. Evidently there is a pronounced hexagonal subcell with parameters $A_1 = A_2 = 2.88 \text{ \AA}$. The complete transformation is:

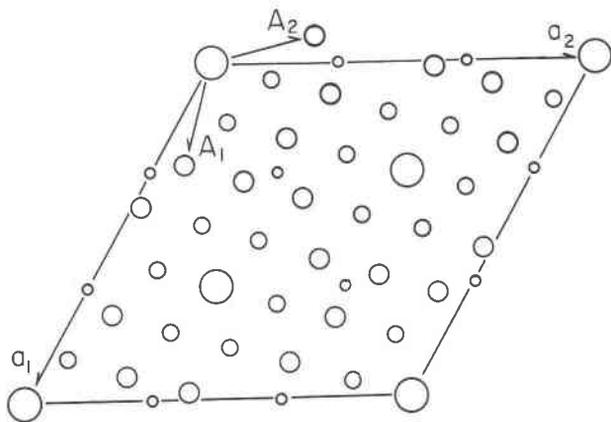


FIG. 1. Representation of the implication function $I_3(xy0)$ with circle radii approximately proportional to peak heights. a_1 and a_2 are the senaite cell translations while A_1 and A_2 are closest-packing subcell translations.

$$\begin{aligned} A_1 &= 4/13a_1 + 1/13a_2 \\ A_2 &= -1/13a_1 + 3/13a_2 \\ C &= 1c \end{aligned} \quad (1)$$

If the space group is $R\bar{3}$ and the crystal is viewed from the other end of the c -axis, the transformation is:

$$\begin{aligned} A_1 &= 3/13a_1 - 1/13a_2 \\ A_2 &= 1/13a_1 + 4/13a_2 \\ C &= 1c \end{aligned} \quad (2)$$

If the space group is $R3$, the two transformations apply to the two enantiomorphs, respectively. In both cases subcell and supercell dimensions are related by $a = A\sqrt{13}$.

The value of the substructure translation in senaite corresponds closely to the oxygen separation of magnetoplumbite. Therefore, senaite is shown to have a structure based on closest-packing, but with an unusual cell translation with respect to it. The substructure is also evident in the zero level c -axis precession photograph which is reproduced in Figure 2.

CHEMICAL COMPOSITION

Table 1 contains four chemical analyses of senaite. Analysis 1 by Hussak and Prior (1898) was determined on material having a specific gravity of 4.78. It shows both ferrous and ferric iron, but Hussak and

TABLE 1. CHEMICAL ANALYSES OF SENAITE

	1	2	3	4
TiO ₂	57.21	52.11	50.32	49.91
Fe ₂ O ₃	20.22	—	—	—
FeO	4.14	26.97	21.99	25.44
PbO	10.51	10.86	9.62	12.01
MnO	7.00	10.42	17.58	11.88
MgO	0.49	0.32	—	—
SnO ₂	0.11	—	—	—
ZrO ₂	—	—	0.84	0.68
Total	99.68	100.68	100.35	99.92
Sp. Gr.	4.78	5.204	5.204	— ^a

^a Sp. Gr. 5.204 assumed.

1. Dattas, Brazil. Hussak and Prior (1898).
2. Dattas, Brazil. Average of two analyses. Hussak and Reitinger (1903).
3. Curralinho, Brazil. Average of two analyses. Hussak and Reitinger (1903).
4. Diamantina, Brazil. Freise (1930).

Reitinger (1903) suggest that this results from contamination of the sample by alteration products, especially limonite. Analyses 2 and 3 were performed on fresh crystals of specific gravity 5.204 and show the presence of only ferrous iron.

In Table 2 are listed the calculated primitive unit cell contents for each of the above analyses. The number of oxygen atoms is evidently 38 or 39. The implication function of Figure 1, $I_3(xy0)$, shows that there are 13 ions in one layer of the closest-packed atom framework. Since the value of c (20.86 Å) corresponds to 9 times the interlayer separation of closest-

TABLE 2. PRIMITIVE UNIT CELL CONTENTS OF SENAITE

	1	2	3	4
Ti	13.48	13.37	12.90	12.80
Fe ³	4.76	—	—	—
Fe ²	1.08	7.69	6.27	7.26
Pb	0.89	1.00	0.88	1.10
Mn	1.86	3.01	5.08	3.43
Mg	0.23	0.16	—	—
Sn	0.01	—	—	—
Zr	—	—	0.14	0.11
Σcations	22.31	25.23	25.27	24.70
Σcations-Pb	21.42	24.23	24.39	23.60
ΣO	38.18	38.60	38.31	37.61

packed oxygen ions ($\sim 2.3 \text{ \AA}$), there must be 9×13 atoms in the framework per hexagonal cell, or 39 per primitive cell.

In magnetoplumbite ($\text{PbFe}_{12}\text{O}_{19}$) the Pb atom is included in the closest-packed framework. The large size of the Pb atom precludes the possibility of it occupying a void in the closest-packed framework without significant distortion. Since no distortion is indicated by the cell parameters and symmetry, all evidence indicates that Pb must occupy the framework in senaite. The calculated unit cell contents indicate the presence of about 24 iron, titanium, manganese, *etc.*, atoms. The suggested formula of senaite is therefore $\text{Pb}(\text{Ti,Fe,Mn,Mg})_{22}\text{O}_{38}$, with $Z=1$ in the primitive cell.

PROGRESS ON ANALYSIS OF THE CRYSTAL STRUCTURE

The structure of senaite has been shown to be based upon a closest-packed anion framework with cations occupying octahedrally and/or tetrahedrally coordinated interstices, except that Pb occupies the framework. The only stacking sequence consistent with a rhombohedral lattice and a 3-layer repeat unit is (2) (1); this fixes the positions of all anion sites relative to one another. If the space group is $R\bar{3}$, the anion coordinates may be readily derived, since the symmetry elements of the anion framework have a unique setting with respect to the symmetry elements of $R\bar{3}$. The three Pb atoms per hexagonal cell can occupy equipoint $3a$ at (0,0,0) or $3b$ at (0,0,1/2). Since the latter is an octahedral void, Pb can only lie on the anion site at the origin.

At the beginning of the structure analysis the space group was assumed to be $R\bar{3}$, since the closest-packed anion framework is at least pseudo-centrosymmetric and a test for pyroelectricity was negative. Three-dimensional intensity data were gathered on the single crystal diffractometer and converted to $|F|_{\text{hkl}}$. These were used to compute the Patterson function $P(uvw)$ and electron density syntheses $\rho(xyz)$. Minimum functions $M_2(xyz)$, derived from $P(uvw)$, and difference Fourierians were also calculated. Interpretation of these functions failed to produce an entirely satisfactory model of the structure, however. The best model gave an R factor of 21.6 percent, after least squares refinement of atomic parameters. In view of this result, it seems probable that the structure is noncentrosymmetric. Even if this is true, however, it is still quite possible that the structure is nearly centrosymmetric and that the trial model is essentially correct.

RELATIONSHIP OF SENAITTE TO DAVIDITE

Pabst (1961) showed that davidite has space group $R3$ or $R\bar{3}$ with $a=10.37 \text{ \AA}$ and $c=20.87 \text{ \AA}$. These values of a and c are equal to those of

senaitite within experimental error. In addition, Pabst published a zero level precession photograph of a twinned, reconstituted metamict crystal. With the exception of reflections due to twinning, this photograph is nearly identical to the corresponding one of senaitite, reproduced in Figure 2. As will be shown later, the powder patterns of these two phases are also similar. Thus, these two phases must be closely related structurally.

Table 3 contains a calculation of the primitive unit cell contents of davidite after Pabst. A compositional similarity between senaitite and davidite is immediately evident. The principal differences of davidite cell

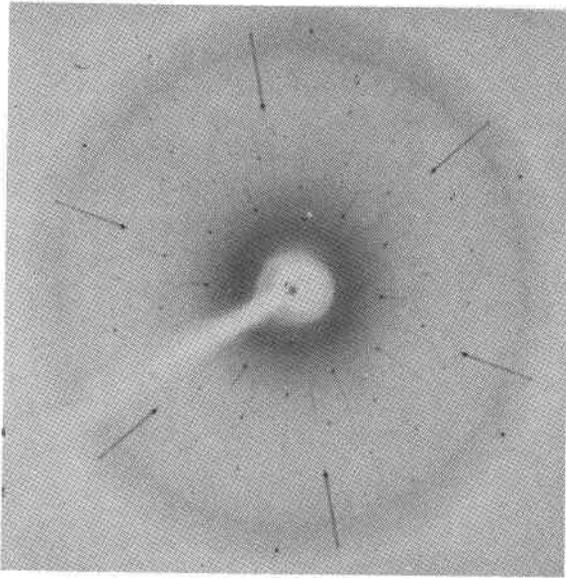


FIG. 2. Zero level, c -axis, $\text{MoK}\alpha$ precession photograph of senaitite from Dattas, Brazil.

contents from those of senaitite are: (1) the absence of lead, (2) the presence of uranium and rare earths and (3) the presence of iron in both oxidation states. The formula suggested by Pabst is $\text{Y}_6\text{Z}_{15}\text{O}_{36}$.

The totals of framework atoms and cations in the davidite formula are both three less than in those of senaitite, for equivalent structural units. By analogy to senaitite, davidite must have either 39 anions per primitive cell (formula $\text{Y}_6\text{Z}_{15}\text{O}_{39}$?) or have a closest-packed structure with anion vacancies. Although apparently not isostructural, senaitite and davidite are at least structurally closely related and based on the same closest-packed sequence.

Pabst reported davidite twinning on $\{52\bar{7}0\}$, with $\{52\bar{7}0\}$, $\{13\bar{4}1\}$, and $\{4\bar{1}\bar{3}2\}$ sets of reciprocal lattice points common to all twin members, and

the strongest on each level. He noted that, "The simplest indices that can be assigned to these sets of points are $\{11\bar{2}0\}$, $\{10\bar{1}1\}$ and $\{01\bar{1}2\}$, corresponding to an hexagonal cell with $a = 2.878$ and $c = 20.87 \text{ \AA}$." The latter cell is the subcell previously defined for senaite. Although twinning seemingly occurs on the high index plane $(52\bar{7}0)$, it occurs on the low index equivalent subcell plane $(11\bar{2}0)$ (by transformation (2)) and thus on a pseudosymmetry element of the substructure. The single-crystal photographs of senaite from Dattas showed no indication of twinning. Photographs of the Santa Quiteria material did have weak extra reflections as a result of twinning on $(25\bar{7}0)$, however. By transformation (1) this is

TABLE 3. CELL CONTENTS OF DAVIDITE FROM PIMA COUNTY, ARIZONA. PABST (1961)

Al	0.13	Mg	0.33
Si	0.09	Ca	—
Ti	11.95	Mn	0.05
V	0.06	Fe ²	1.80
Cr	0.21	Sr	0.05
Fe ³	4.27	Pb	—
	—	Zr	0.08
Σ'	16.71	Ce ^a	0.99
		Th	0.03
		U	0.24
		Na	0.01
		K	0.03
			—
		Σ''	3.61
		$\Sigma(\Sigma' + \Sigma'')$	20.32
		0	35.44

^a Rare earths treated as Ce₂O₃.

equivalent to $(11\bar{2}0)$, further substantiating the relationship with davidite.

The crystal chemical relationships of davidite have been further investigated by Contag and Strunz (1961) and Contag (1962). They describe oriented intergrowths of rutile and davidite in which $[100]$ of rutile parallels $[0001]$ of davidite and $[001]$ of rutile parallels $[4\bar{1}30]$ (subcell $[1000]$) of davidite. The formula $\text{La}_{0.5}(\text{U,Ce})_{1.0}(\text{Fe}^{+2}, \text{Fe}^{+3})_{4.5}(\text{Ti}, \text{Fe}^{+3})_{15}\text{O}_{36}$ is derived. The structure is recognized as being based on oxygen closest-packing with three (U,Ce) atoms situated on anion sites in the framework. It is evident that these large cations play the same role that lead does in the senaite structure. A close structural relationship among davidite, crichtonite, mohsite, and senaite is suggested on the basis of their chemical and morphological similarities.

RELATIONSHIP OF SENAITE TO OTHER CLOSEST-PACKED MINERALS

Berry (1951) has determined the unit cell parameters of magnetoplumbite as $a=5.88$ and $c=23.02$ Å. It has the idealized formula $\text{PbFe}_{12}\text{O}_{19}$ with $Z=2$ and space group $P6_3/mmc$. Berry reported the unit cell contents $\text{Pb}_2(\text{Fe}_{15}\text{Mn}_7\text{AlTi})\text{O}_{38}$ for magnetoplumbite from the type locality. Hibonite $(\text{Ca}, \text{Y} \dots)(\text{Al}, \text{Ti}, \text{Si}, \text{Mg})_{12}\text{O}_{19}$ (Currien *et al.*, 1956) is isostructural with magnetoplumbite as are several synthetic compounds such as $\text{BaFe}_{12}\text{O}_{19}$. Plumboferrite has been assigned the formula PbFe_4O_7 (Johansson, 1928). It is certainly closely related chemically to magnetoplumbite, but its exact formula and crystal structure are uncertain. A crystal chemical study of this mineral is now in progress.

It is clear that magnetoplumbite, senaite, plumboferrite, and davidite form a related group of phases. All are complex multiple oxides of iron and/or titanium with a small number of large metal cations. All are based on oxygen closest-packing with the large cations occupying anion sites in the framework. Thus, senaite provides the structural link between the davidite and magnetoplumbite groups.

Several other minerals which are chemically unrelated to senaite but which have similar unit cell geometry have recently been described. Benstonite (Lippmann, 1962*a*) has the formula $\text{Ca}_7\text{Ba}_6(\text{CO}_3)_{13}$. It has a superstructure-substructure relationship to calcite which is exactly analogous to that of senaite to an oxygen closest-packed framework. Although the anion configuration of benstonite is not closest-packed, each cation layer consists of a closest-packed array of 13 atoms. The superstructure is caused by cation ordering within cation layers. Benstonite has space group $R\bar{3}$ or $R\bar{3}$ and supercell dimensions $a=18.28$ and $c=8.67$ Å. These are related to the subcell (calcite cell) dimensions A and C by $a=A\sqrt{13}$ and $c=C/2$.

The relationship between benstonite and klockmannite has been described by Lippmann (1962*b*) and Takeda and Donnay (1965). Klockmannite (CuSe) has the probable space group $P6_3/m$ and cell parameters $a=14.26$ $c=17.25$ Å. Its structure is a derivative of the structure of covellite (CuS). It has a substructure-superstructure relation identical to that described for senaite and benstonite. Taylor and Underwood (1960) suggest that the mineral is twinned on $(11\bar{2}0)$, which is the substructure twin plane of senaite and davidite.

This relationship between substructure and superstructure can be interpreted as one of the simpler compound tessellations, as pointed out by Takeda and Donnay (1965). The particular tessellation is of the type $\{3, 6\} [n \{3, 6\}]$ with $(u, v) = (1, 4)$ and $n=13$; $n=u^2-uv+v^2$ and is the multiplicity of the supercell with respect to the subcell. Ranganathan

TABLE 4. POWDER PATTERNS OF SENAIITE AND DAVIDITE^a

Senaite				Davidite (Pabst 1961)				
<i>hkil</i>	<i>d_{calc}</i>	<i>d_{obs}</i>	<i>I</i>	<i>hkil</i>	<i>d_{calc}</i>	<i>d_{obs}</i>	<i>I</i>	
10 $\bar{1}$ 1	8.28	8.27	20	000·3	6.96	6.86	18	
				11 $\bar{2}$ 0	5.18	5.21	15	
11 $\bar{2}$ 3	4.17	4.17	25	11 $\bar{2}$ 3	4.16	4.18	24	
01 $\bar{1}$ 5	3.79	3.78	20	01 $\bar{1}$ 5	3.79	3.81	7	
02 $\bar{2}$ 4	3.41	3.43	90	02 $\bar{2}$ 4	3.40	3.42	72	
				21 $\bar{3}$ 1	3.35	3.36	8	
12 $\bar{3}$ 2	3.24	3.25	5	12 $\bar{3}$ 2	3.228	3.21	20	
20 $\bar{2}$ 5	3.06	3.04	20	20 $\bar{2}$ 5	3.057	3.065	50	
30 $\bar{3}$ 0	3.01			30 $\bar{3}$ 0	2.994	3.000	40	
11 $\bar{2}$ 6	2.892			2.894	100	11 $\bar{2}$ 6	2.889	2.895
				21 $\bar{3}$ 4	2.845	2.850	60	
30 $\bar{3}$ 3	2.761	2.763	25	30 $\bar{3}$ 3	2.748	2.755	22	
12 $\bar{3}$ 5	2.641	2.644	20	12 $\bar{3}$ 5	2.633	2.640	33	
				22 $\bar{4}$ 0	2.593	2.595	18	
13 $\bar{4}$ 1	2.485	2.468	50	13 $\bar{4}$ 1	2.473	2.480	38	
22 $\bar{4}$ 3	2.440			22 $\bar{4}$ 3	2.429	2.429	25	
30 $\bar{3}$ 6	2.275							
20 $\bar{2}$ 8	2.258	2.265	50	13 $\bar{4}$ 4	2.248	2.248	50	
				31 $\bar{4}$ 5	2.139	2.139	40	
31 $\bar{4}$ 5	2.146	2.146	50	12 $\bar{3}$ 8	2.068	2.072	5	
				1·0· $\bar{1}$ ·10	2.033	2.029	5	
04 $\bar{4}$ 5	1.984	1.981	75	04 $\bar{4}$ 5	1.977	1.982	12	
				32 $\bar{5}$ 4	1.916	1.919	25	
13 $\bar{4}$ 7	1.917	1.914	5					
				0·2· $\bar{2}$ ·10	1.893	1.897	5	
0·1· $\bar{1}$ ·11	1.856	1.852	5	23 $\bar{5}$ 5	1.847	1.849	10	
23 $\bar{5}$ 5	1.855			31 $\bar{4}$ 8	1.801	1.803	38	
31 $\bar{4}$ 8	1.806			1.804	40	2·1· $\bar{3}$ ·10	1.778	1.780
41 $\bar{5}$ 6	1.714	1.713	40	000·12	1.739	1.741	<5	
1·2· $\bar{3}$ ·11	1.657	1.656	5	41 $\bar{5}$ 6	1.707	1.708	22	
50 $\bar{5}$ 5								
1·3· $\bar{4}$ ·10	1.602	1.603	70	1·1· $\bar{2}$ ·12	1.649	1.652	12	
15 $\bar{6}$ 2						1·3· $\bar{4}$ ·10	1.600	1.501
				42 $\bar{6}$ 5	1.572	1.572	<5	
33 $\bar{6}$ 6	1.554	1.551	20	33 $\bar{6}$ 6	1.548	1.548	10	
				3·1· $\bar{4}$ ·11	1.509	1.509	15	
3·1· $\bar{4}$ ·11	1.511	1.508	20	3·2· $\bar{5}$ ·10	1.466	1.467	5	
15 $\bar{6}$ 5	1.511							
3·0· $\bar{3}$ ·12	1.505							
				5270	1.438	1.439	40	

^a CuK α radiation, Ni filter. Camera diameter 114.59 mm.

TABLE 4—(continued)

Senaite				Davidite (Pabst 1961)			
<i>hkl</i>	d_{calc}	d_{obs}	<i>I</i>	<i>hkl</i>	d_{calc}	d_{obs}	<i>I</i>
2·2·4·12	1.446	1.447	70			1.419	8
5270	1.445						
5273	1.415	1.415	10			1.376	10
2·0·2·14							1.360
6066	1.380	1.380	10			1.346	8
						1.302	6
1·1·2·15	1.344	1.344	2			1.275	<5
		1.305	2			1.246	5
		1.248	2				
		1.218	2				
		1.189	2				
		1.154	2				
		1.127	10				
		1.104	10				

(1966) treats such relationships for a cubic lattice using the concept of coincidence-site lattices. Although the above minerals differ widely in chemistry and crystal structure, they are all based on variations of the same underlying theme of closest-packing.

SENAITE POWDER PATTERN

Table 4 gives indexed powder data for senaite from Dattas and for davidite (Pabst, 1961). Material for the senaite pattern was carefully hand sorted from the specimen used as a source for single crystal material. Interplanar spacings for senaite were calculated using an IBM 7090 program. All observed powder lines are accounted for by calculated values. A comparison of the patterns of senaite and davidite shows that there is nearly complete correspondence of observed interplanar spacings. Although a rough correlation exists between the intensities, there are marked differences for some reflections. This is not unexpected in view of the chemical differences between the two minerals. Each spacing may be represented by two indices corresponding to the different orientations of the cell. To facilitate comparison with the pattern of davidite, the indices chosen are those given by Pabst for corresponding spacings in davidite.

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