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The crystal structure of viitaniemiite

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Abstract

The crystal structure of viitaniemiite $Na(Ca,Mn)AlPO_4F_2OH$ $a = 5.457(2)$, $b = 7.151(2)$, $c = 6.836(2)$Å, $\beta = 109.36(3)°$ $V = 251.68$Å^3, $Z = 2$, space group $P2_1/m$, has been solved by Patterson and Fourier methods and refined by the least-squares method to an R index of 0.037 for 728 observed ($>2\sigma$) reflections. The structure contains two sets of infinite chains parallel to the b-axis, one composed of $AlO_2(OH)_2F_2$ octahedra sharing opposite OH corners and the other of $(Ca,Mn)O_4F_2$ octahedra sharing opposite O–O edges. These chains alternate laterally sharing F corners to form a set of parallel sheets held together by PO_4 tetrahedra and NaO_4F_4 gable disphenoids.

The sheet structure of viitaniemiite containing octahedrally coordinated atoms in two separate positions resembles that of montebrasite and eosphorite. These three related phosphate minerals are associated with each other in the type locality of viitaniemiite, Viitaniemi pegmatite, Orivesi, southern Finland, where they crystallized during hydrothermal replacement processes caused by residual fluids of the pegmatite melt.

Introduction

Viitaniemiite occurs as a rare hydrothermal mineral in the phosphate-rich Viitaniemi pegmatite, Orivesi, southern Finland. One of the authors (SIL) has described it as a new mineral in a study on the mineralogy and petrology of the granitic pegmatities of the Eräjärvi area (Lahti, 1981). The mineral was encountered as an inclusion in eosphorite aggregate and is associated with morinite, another aluminum-bearing phosphate mineral. Structure analysis confirmed the ideal formula $Na(Ca,Mn)AlPO_4F_2OH$ with $Z = 2$, although wet chemical analysis indicates that some of the fluorine may be replaced by OH groups. The crystal data measured during the structure analysis are given in Table 1. The X-ray powder data, the optical properties, the chemical data, and the mineralogical description of the mineral have been given by Lahti (1981) and are not reproduced here. A preliminary description of the structure has been given by Lahti and Pajunen (1982). To date viitaniemiite has been identified positively in only three localities: at Viitaniemi, in museum specimens collected from druses of granite in Greifenstein, Sachsen (East Germany), and from Francon quarry, northeastern Montreal, where its occurrence has been confirmed by Ramik et al. (1983). In this quarry viitaniemiite was encountered as very small crystals in vesicles of silicocarbonatite together with cryolite, calcite, quartz, and weloganite. Because the powder diffraction data of viitaniemiite resemble those of an unnamed mineral from Greifenstein, Sachsen, given on JCPDS card 13-0587, the present authors studied several museum specimens taken from this locality and identified viitaniemiite from the samples labeled as lacroixite (cf. Mrose, 1971). According to the descriptins by Slavik (1914, 1915), lacroixite (and therefore also viitaniemiite) occurs there in druses of lithiogranite together with ježekite (sodian morinite), apatite, childrenite (Fe end member of the isomorphous series childrenite-eosphorite), roscherite, and tourmaline. Preliminary microanalyzer determinations showed that Greifenstein viitaniemiite is rich in calcium and contains only a few percent of manganese; the fluorine content, however, is equal to that of the Finnish viitaniemiite. Due to its different chemical composition, the Greifenstein viitaniemiite has a larger unit cell ($a = 5.48$Å, $b = 7.18$Å, $c = 6.85$Å, $\beta = 109.00°$, $V = 254.84$Å^3; based on the precession films from sample no. 86746 in Harvard Mineralogical Museum). Detailed studies on the Greifenstein viitaniemiite and lacroixite are in progress, because some of the data cited for lacroixite obviously derive from viitaniemiite.

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Table 1. Crystal data for viitaniemiite

a = 5.457(2) Å	V = 251.68 $Å^3$
b = 7.151(2) Å	Z = 2
c = 6.836(2) Å	D(calc.) = 3.242 g/cm^3
β = 109.36(3)°	D(meas) = 3.245 g/cm^3
Space group $P2_1/m$	μ(Mo$K\alpha$) = 2.42 cm^{-1}
Formula (ideal) $Na(Ca,Mn)AlPO_4F_2OH$	

Experimental

For the structure analysis a transparent crystal plate of viitaniemiite measuring about 0.1 × 0.2 × 0.3 mm was removed from the type material. Roundish crystals suitable for study are difficult to obtain, because the mineral forms thin, sometimes radial, crystal plates (0.02–0.2 mm thick) flattened parallel to the $(10\bar{1})$ plane.

Single crystal X-ray precession photographs of this crystal exhibit monoclinic symmetry with systematic absences $0k0$, $k = 2n + 1$ consistent with the space groups $P2_1/m$ and $P2_1$. The unit cell dimensions (298 K) given in Table 1 were obtained by least-squares refinement of angular settings of 20 reflections centered on a Nicolet P3 automatic four-circle diffractometer using graphite monochromated Mo$K\alpha$ radiation (λ = 0.7107Å). A unique set of intensities was measured using the ω-scan method with variable scan speed. Of the 788 reflections with $3° < 2\theta < 50°$, 728 were regarded as observed, their intensity being larger than 2.0 times the estimated standard deviation. Three standard reflections measured for every 50 reflections showed no systematic variation. The intensity data were corrected for Lorentz and polarization effects and an empirical absorption correction based on the φ-scan method was applied (North et al., 1968), with the coefficient ranging from 0.707 to 1.0.

Structure determination

The space group, assumed to be $P2_1/m$ on the basis of diffraction intensity statistics, was later confirmed by the structure solution. The symmetry in the space group $P2_1/m$ requires that most of the atoms be in special positions. The structure was solved by Patterson and Fourier methods. The Patterson synthesis showed that most of the atoms are situated at $y = \pm 0.25$. A solution was found for Al and (Ca,Mn) in the centers of symmetry, for F and one O in general positions and for other atoms on the mirror plane.

The atomic scattering factors used were those of Cromer and Mann (1968) for non-hydrogen atoms with anomalous dispersion coefficients from the *International Tables for X-ray Crystallography* (Ibers and Hamilton, 1974), and of Stewart et al (1965) for hydrogen. The structure was refined by the full-matrix least-squares method. The function minimized was $\Sigma w \| F_o| - |F_c\|^2$ with unit weights. $R = 0.071$ was obtained by refinement with isotropic temperature factors for non-hydrogen atoms. The temperature factors were converted to anisotropic in the form given in Table 2; refinement reduced R to 0.057. A difference map calculated at this stage revealed a peak on the mirror plane 1Å from O(4). This peak was assumed to be the hydrogen atom. The hydrogen atom was included in the refinement with a fixed isotropic temperature factor $U = 0.06Å^2$.

The final refinement included positional parameters, anisotropic thermal parameters, and occupancy factors for calcium and manganese. The initial values for the occupancy factors were obtained from chemical analysis. The occupancy factors converged to 0.592(5) for calcium and 0.401(4) for manganese. Final R was 0.037, $R_w = 0.043$ and $S = (\Sigma w \Delta F^2/m-n)^{1/2} = 4.12$. The difference map had its largest electron density 0.61 $eÅ^{-3}$ in the vicinity of the (Ca,Mn) atom. The calculations were performed on a Univac 1108 computer using programs of the XRAY76 system (Stewart, 1976). The final atomic positions and thermal parameters are listed in Table 2. The interatomic distances and bond angles are reported in Table 3. Table 4, giving observed and calculated structure factors, is on deposit at the Business Office of the Mineralogical Society of America.[1]

Description of the structure

A stereoscopic view of the structure is presented in Figure 1. Al and (Ca,Mn) are in the centers of symmetry, phosphorus and two of the oxygen atoms of the phosphate ion are situated on the mirror plane. The sodium and hydroxyl ions are also on the mirror plane. Fluorine and one oxygen of the phosphate ion are in general positions.

Aluminum is surrounded by two oxygens, two hydroxyls and two fluorines in octahedral arrangement. The $AlO_2(OH)_2F_2$ octahedra form chains parallel to the **b**-axis by sharing opposite OH^- corners. In this 7Å corner-sharing OH–Al–OH–Al chain (Moore, 1980) the OH bridges are in trans configuration. (Ca,Mn) is surrounded by four oxygens and two fluorines in distorted octahedral arrangement. The octahedra share opposite O–O edges to form a set of chains parallel to the **b**-axis. The shared edge is considerably shorter than the others, suggesting repulsion between neighboring (Ca,Mn) cations. The two chains alternate by sharing F corners to form a set of parallel sheets. Adjacent sheets are held together by phosphate ions. The phosphate ion is a nearly ideal tetrahedron with O–P–O angles in the range 108.3–112.2°. Additional linkage between the sheets is provided by the NaO_4F_4 polyhedron (Fig. 2). Five Na–O and Na–F distances are between 2.3–2.5Å, defining a distorted square pyramid, but three additional distances of 2.8Å are included, resulting in a polyhedron with coordination number eight. The next larger distances are >3.1Å. This polyhedron, gable disphenoid, has been discussed in detail by Moore (1981). Previously it has only been found in wyllieite (Moore and Molin-Case, 1974) and fillowite (Araki and Moore, 1981).

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[1] To obtain a copy of Table 4, order Document Am-84-251 from the Mineralogical Society of America, Business Office, 2000 Florida Avenue, N. W., Washington, D. C. 20009. Please remit $5.00 in advance for the microfiche.

Table 2. Atomic coordinates and anisotropic thermal vibration parameters for viitaniemiite

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
(Ca,Mn)	0.5	0.0	0.5	0.0172(9)	0.0099(9)	0.0094(8)	-0.0011(8)	0.0048(6)	-0.0009(6)
Al	0.0	0.0	0.0	0.0125(6)	0.0076(6)	0.0068(5)	-0.0006(5)	0.0029(4)	-0.0003(5)
Na	0.4675(5)	0.25	-0.0463(4)	0.0275(13)	0.0235(12)	0.0387(15)	0.0	0.0298(12)	0.0
P	-0.0366(2)	0.25	0.3683(2)	0.0117(5)	0.0102(5)	0.0066(5)	0.0	0.0041(4)	0.0
F	0.3468(4)	0.0030(3)	0.1547(3)	0.0137(9)	0.0160(10)	0.0154(9)	-0.0001(8)	0.0008(7)	0.0031(9)
O(1)	0.2456(7)	0.25	0.5160(17)	0.0133(17)	0.0370(24)	0.0136(17)	0.0	0.0046(14)	0.0
O(2)	-0.2336(7)	0.25	0.4860(5)	0.0151(16)	0.0188(16)	0.0080(14)	0.0	0.0067(13)	0.0
O(3)	-0.0850(6)	0.0755(4)	0.2300(4)	0.0401(16)	0.0083(11)	0.0168(12)	-0.0046(11)	0.0194(11)	-0.0010(10)
O(4)	0.0239(7)	0.25	-0.0815(5)	0.0191(16)	0.0076(14)	0.0063(14)	0.0	0.0052(12)	0.0
H	-0.005(19)	0.25	-0.201(15)	U=0.06					

The values of x, y, z are given in fractional coordinates, the anisotropic temperature factor is of the form $\exp(-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{12}a^*b^*hk + 2U_{13}a^*c^*hl + 2U_{23}b^*c^*kl))$. Estimated standard deviations in parentheses refer to the last digits.

There is only one hydrogen bond in the structure: the hydrogen atom of the hydroxyl ion is bonded to the phosphate oxygen atom O(2) with hydrogen bond parameters 2.09(9)Å and 157(10)°.

Comparison with related structures

Viitaniemiite is structurally and chemically related to two aluminum-bearing hydroxylated phosphate minerals: amblygonite–montebrasite and eosphorite–childrenite. It is interesting to note that these three minerals are intimately associated in the type locality of viitaniemiite, montebrasite being crystallized before viitaniemiite, and eosphorite after it. Figure 3 shows polyhedral representations of the three structures taken from a certain plane to compare them with each other. The base structure of both viitaniemiite and eosphorite (see the structure determination by Hanson, 1960, and the sketch by Moore, 1970) contains octahedral corner-sharing and edge-sharing chains with a 7Å repeat. The chains are parallel to the **b**-axis of viitaniemiite and the **c**-axis of eosphorite. The

Table 3. Interatomic distances and angles for viitaniemiite

(Ca,Mn) - O(1)	2.288(3)	O(1) - (Ca,Mn) - $O(2)^{i}$	78.4(1)	O(1) - $O(2)^{i}$	2.916(6)
(Ca,Mn) - $O(2)^{i}$	2.326(3)	O(1) - (Ca,Mn) - F	91.3(1)	O(1) - F	3.230(5)
(Ca,Mn) - F	2.228(2)	$O(2)^{i}$ - (Ca,Mn) - F	88.2(1)	$O(2)^{i}$ - F	3.171(3)
Al - O(3)	1.861(3)	O(3) - Al - O(4)	92.0(1)	O(3) - O(4)	2.699(5)
Al - O(4)	1.890(1)	O(3) - Al - F	90.5(1)	O(3) - F	2.625(4)
Al - F	1.837(2)	O(4) - Al - F	90.0(1)	O(4) - F	2.635(3)
Na - $O(1)^{ii}$	2.832(5)	$O(1)^{ii}$ - Na - O(4)	80.1(1)	$O(1)^{ii}$ - O(4)	3.354(6)
Na - $O(3)^{i}$	2.834(4)	$O(1)^{ii}$ - Na - F^{iii}	75.0(1)	$O(1)^{ii}$ - F^{iii}	3.156(4)
Na - O(4)	2.352(5)	$O(3)^{i}$ - Na - $O(3)^{iv}$	52.2(1)	$O(3)^{i}$ - $O(3)^{iv}$	2.496(4)
Na - F	2.458(3)	$O(3)^{i}$ - Na - F	69.1(1)	$O(3)^{i}$ - F	3.015(4)
Na - F^{iii}	2.312(3)	$O(3)^{i}$ - Na - F^{iii}	59.8(1)	$O(3)^{i}$ - F^{iii}	2.604(3)
		O(4) - Na - F	66.4(1)	O(4) - F	2.635(3)
		F - Na - F^{iii}	81.0(1)	F - F^{iii}	3.100(4)
		F - Na - F^{v}	91.9(1)	F - F^{v}	3.533(4)
		F^{iii} - Na - F^{vi}	103.0(2)	F^{iii} - F^{vi}	3.618(4)
P - O(1)	1.536(4)	O(1) - P - O(2)	112.2(2)	O(1) - O(2)	2.554(6)
P - O(2)	1.542(5)	O(1) - P - O(3)	109.6(1)	O(1) - O(3)	2.509(4)
P - O(3)	1.535(3)	O(2) - P - O(3)	108.3(1)	O(2) - O(3)	2.494(5)
		O(3) - P - $O(3)^{v}$	108.8(2)	O(3) - $O(3)^{v}$	2.496(4)
O(4) - H	0.78(10)	O(4) - H ...$O(2)^{ii}$	157(10)	H ...$O(2)^{ii}$	2.09(9)

Symmetry code (i) = 1 + x, y, z (ii) = x, y, -1 + z (iii) = 1 - x, -y, -z
(iv) = 1 + x, ½ - y, z (v) = x, ½ - y, z (vi) = 1 - x, ½ + y, -z

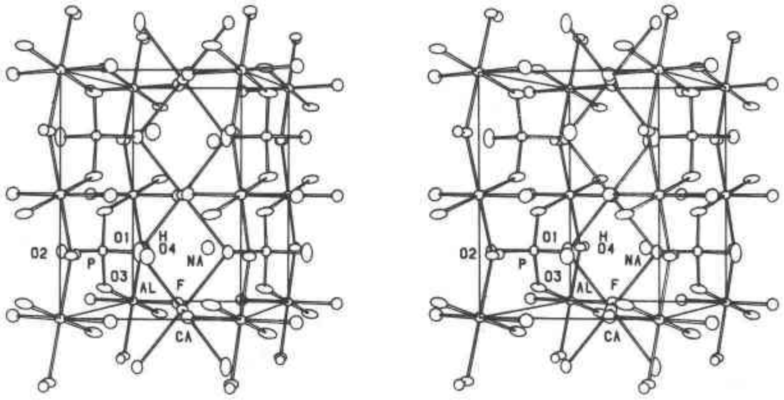

Fig. 1. Stereoscopic view of the viitaniemiite structure. The origin is at the lower left-hand corner, **a** is horizontal, **b** is vertical and **c** towards the viewer.

edge-sharing (Ca,Mn)O_4F_2 octahedra of the viitaniemiite structure are replaced by (Mn,Fe)$O_4(OH)_2$ octahedra in the eosphorite structure, and the corner-sharing $AlO_2(OH)_2F_2$ octahedra by $AlO_2(OH)_2(H_2O)_2$ octahedra. The chains alternate laterally sharing F corners in viitaniemiite and OH corners in eosphorite. The octahedral cations are situated on the same plane and form a set of sheets parallel to {010} in viitaniemiite and {001} in eosphorite. Due to the different linkage of the octahedral chains, the structure of viitaniemiite is monoclinic with $\beta \approx 109°$, whereas that of eosphorite is pseudoorthorhombic (monoclinic with $\beta = 90°$) and the unit cell about four times larger.

The (100) section of the amblygonite structure also has many features in common with the {010} section of viitaniemiite. The α-angle of amblygonite ($\approx 112°$) is nearly identical to the β-angle of viitaniemiite ($\approx 109°$), and the lengths of the crystallograpnic axes on these planes are close to each other. According to the structure determination by Baur (1959) the amblygonite structure contains $AlO_4(F,OH)_2$octahedra, the central atom Al being in two positions, Al(1) in 0,0,0 and Al(2) in 0,0.5,0.5 corresponding to Al and (Ca,Mn) in the viitaniemiite structure. Amblygonite is, however, triclinic and the unit cell contains only one sheet. The PO_4 tetrahedra, the $LiO_4(OH,F)$ polyhedra in amblygonite and, replacing it, the NaO_4F_4 gable disphenoids in viitaniemiite hold the neighboring sheets together.

Linked together by F^- ions, the Al octahedra and (Ca,Mn) octahedra of viitaniemiite form a chain of corner-sharing polyhedra parallel to the $(10\bar{1})$ plane. Similar chains can also be seen in the amblygonite structure, but the Al octahedra share $(F,OH)^-$ instead of F^- alone. F^- may also be substituted by $(OH)^-$ in viitaniemiite, because the structure determination indicates an F/OH ratio 2/1, whereas the chemical formula based on the wet chemical analysis of the powdered sample shows a much lower F/OH ratio, 1.18.

Mineralogical studies show that the perfect cleavage plane follows the {100} plane in eosphorite and the $(10\bar{1})$ plane in viitaniemiite. In addition, amblygonite-montebrasite has a prominent cleavage parallel to $(0\bar{1}1)$. The cleavage plane of eosphorite follows the "hole" direction of the structure between the Al octahedra (see Fig. 3), and similarly the direction of the large alkali metal polyhedra (or the chain direction) in the other structures.

Progressive hydrothermal leaching and replacement of primary phosphates produced abundant hydrated or hydrous secondary phosphates during the crystallization of the Viitaniemi pegmatite dike. The number of (Mn,Fe) phosphates is particularly high, but various Al- and Be-bearing secondary phosphates are also known in the deposit (Volborth, 1954; Lahti, 1981). In the type locality viitaniemiite is closely associated with amblygonite–montebrasite, eosphorite, morinite, crandallite, and apatite. The above phosphate minerals occur in the sugar albite and cleavelandite replacement units and fracture fillings which were formed as final crystallization products of the

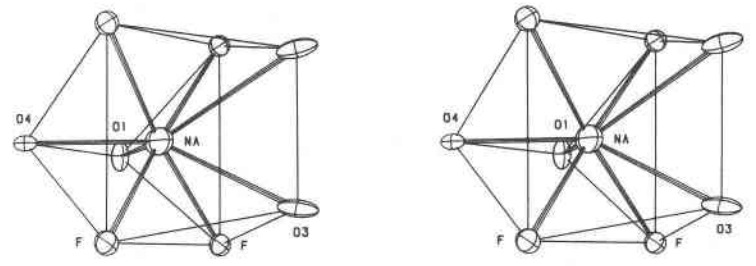

Fig. 2. Stereoscopic view of the NaO_4F_4 polyhedron (gable disphenoid) in the viitaniemiite structure.

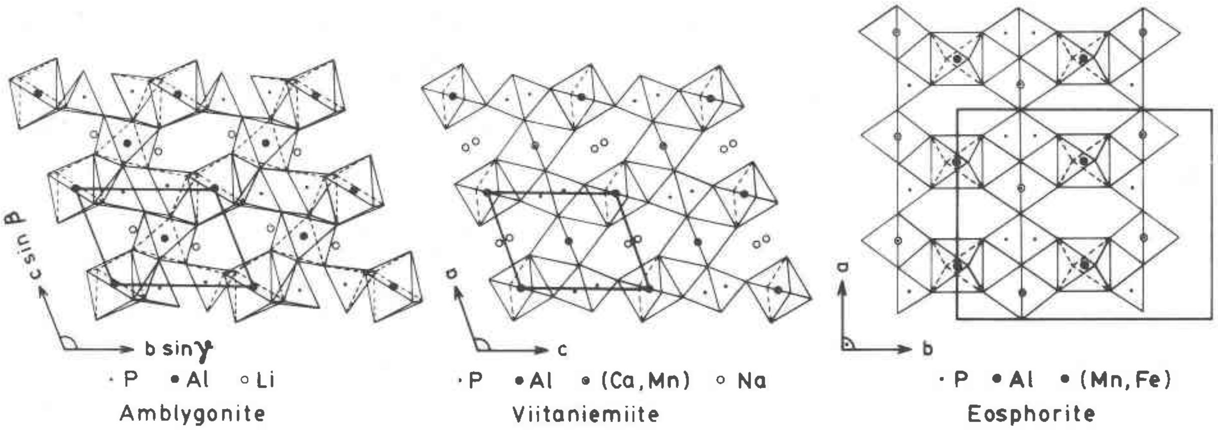

Fig. 3. Polyhedral representations of the structure of amblygonite, eosphorite and viitaniemiite.

volatile-rich pegmatite melt. The viitaniemiite sample studied in detail occurred as an inclusion in eosphorite between montebrasite crystal plates. Eosphorite replaces montebrasite, which is strongly altered. Viitaniemiite crystallized after montebrasite, but before eosphorite, crandallite and apatite. The hydrothermal residual solutions rich in Na^+, Mn^{2+}, Ca^{2+}, F^- and OH^- reacted with amblygonite-montebrasite, dissolving it or only partly replacing the structure. The basic structure of amblygonite is therefore closely related to that of viitaniemiite and eosphorite which crystallized later. On the other hand, the structure of morinite, which crystallized before viitaniemiite, contains corner-sharing octahedral dimers with a composition of $Al_2F_4(OH)_2O_4$ linked with two PO_4 tetrahedra to a complex polyhedral cluster (Hawthorne, 1979). Condensation of octahedral dimers can also produce similar octahedral chains with a 7Å repeat, as in viitaniemiite and eosphorite. Crandallite, which crystallized with apatite as the last phosphate mineral in a solution cavity of viitaniemiite, has an alunite-derivative sheet structure. As shown by Moore (1980), this structure can also be conceived as progressive condensation of octahedral corner-sharing chains bridged by OH^- in the trans configuration.

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