

Johninnesite: Crystal-structure determination and its relationship to other arsenosilicates

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ABSTRACT

Johninnesite, $\text{Na}_2\text{Mn}_3^{2+}(\text{Mg}, \text{Mn}^{2+})_7(\text{OH})_8[\text{AsO}_4]_2[\text{Si}_6\text{O}_{17}]_2$, from the Kombat mine, Namibia, is triclinic, $P\bar{1}$ with $a = 10.485(8)$, $b = 11.065(7)$, $c = 9.654(6)$ Å, $\alpha = 107.11(4)^\circ$, $\beta = 81.17(5)^\circ$, $\gamma = 111.86(4)^\circ$, and $Z = 1$. The crystal structure was solved by direct methods and refined to $R = 7.5$ and $R_w = 6.4\%$. Of the 6125 measured reflections, 1030 were removed because of twinning overlap.

The structure is layered with a tetrahedral layer consisting of independent AsO_4 and Si_6O_{17} chains, an octahedral layer of edge-sharing $(\text{Mn}, \text{Mg})\text{O}_6$ octahedra with one-sixth of the octahedral sites vacant, and a layer with single MnO_6 octahedral chains separated by NaO_6 distorted octahedra. The structure does not resemble those of any of the other 13 arsenosilicates, but the layering and silicate chains resemble those of the howieite structure.

INTRODUCTION

Johninnesite, first described by Dunn et al. (1986), is a sodium manganese arsenosilicate from the Kombat mine, Namibia. It occurs as light-yellowish brown, bladed to fibrous aggregates up to 4 cm across. The mineral was reported to be triclinic and having unit-cell contents $\text{Na}_{2.06}\text{Mg}_{4.20}\text{Fe}_{0.03}\text{Mn}_{11.83}\text{As}_{1.90}\text{Si}_{12.18}\text{O}_{43.22}(\text{OH})_{5.95}$ (Dunn et al., 1986). The authors stated in their original description that a crystal structure analysis was necessary to determine the specific valence of Mn and As and to determine whether the H occurs as OH or H_2O . The present study answers these questions and elucidates the crystal chemistry of johninnesite.

EXPERIMENTAL METHODS

Several crystal fragments of type johninnesite (NMNH 163207) were selected for X-ray precession photographs to check diffraction spot quality. Crystals are elongated along [001] and flattened on {100}. The fibrous nature of the mineral gives rise to split diffraction maxima. The crystal chosen for the intensity data collection measured $0.37 \times 0.13 \times 0.07$ mm. It was mounted on a Nicolet R3m automated four-circle diffractometer. The experimental settings and structure refinement data are given in Table 1. Twenty-five reflections were used to orient the crystal and refine the cell parameters (Table 1), which are in good agreement with those of Dunn et al. (1986). The intensity data collection of one asymmetric unit of the reciprocal cell in the region $4\text{--}60^\circ 2\theta$ ($\text{MoK}\alpha$ radiation) consisted of 5807 unique reflections.

Reduction of the intensity data and solution and refinement of the structure were done by the SHELXTL package of programs (Sheldrick, 1990). Data reduction

included background, scaling, Lorentz and polarization corrections, and absorption corrections. For the absorption correction, 11 intense diffraction maxima over the 2θ range $10\text{--}54^\circ$ were chosen for ψ diffraction-vector scans after the method of North et al. (1968). The ψ scan intensity data were used to refine an ellipsoidal, empirical absorption correction. Absorption correction reduced the merging R of the ψ scan data from 12.4 to 5.0%. Scattering curves for neutral atoms from Cromer and Mann (1968) and anomalous dispersion coefficients from Cromer and Liberman (1970) were used in the structure determination.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved by direct methods. The mean value of $|E^2 - 1|$ is 1.04, which favors the centrosymmetric space group $P\bar{1}$ for the triclinic crystal system. The solution with the best combined figure of merit showed 39 possible atom peaks, which were assigned scattering curves on the basis of peak heights and coordinations. This initial model refined to give a residual index $R =$

TABLE 1. Structure determination data for johninnesite

Unit-cell contents: $\text{Na}_2\text{Mn}_3(\text{Mg}, \text{Mn})_7(\text{OH})_8[\text{AsO}_4]_2[\text{Si}_6\text{O}_{17}]_2$, $Z = 1$	
Space group: $P\bar{1}$	
$a = 10.485(8)$ Å	$\alpha = 107.11(4)^\circ$
$b = 11.065(7)$ Å	$\beta = 81.17(5)^\circ$
$c = 9.654(6)$ Å	$\gamma = 111.86(4)^\circ$
Radiation/Mono = Mo/graphite at 50 kV, 35 mA	
$\mu = 5.78 \text{ mm}^{-1}$; minimum and maximum transmission = 0.201/0.421	
No. of $F_o = 4904$; no. of $F_o > 6\sigma = 3183$	
Final $R = 7.5\%$; final $R_w = 6.3\%$	
$R = \Sigma (F_o - F_c) / \Sigma F_o$	
$R_w = [\Sigma w(F_o - F_c) / \Sigma wF_c^2]^{1/2}$; $w = \sigma^{-2}(F_o)$	

TABLE 2. Positional coordinates ($\times 10^4$), displacement parameters ($\times 10^4 \text{ \AA}^2$), and bond valence sums (e) for johninnesite

Site	x	y	z	U_{eq}	BVS
Na1	5148(6)	6095(5)	2781(7)	43(2)	0.87
Mn1	3833(2)	444(1)	4424(2)	22(1)	1.85
Mn2	6091(2)	9604(1)	2075(2)	23(1)	1.93
Mn3	3620(2)	309(1)	1115(2)	24(1)	1.94
Mn4	100(2)	7729(1)	4284(2)	23(1)	2.04
Mn5	0	0	0	28(1)	2.20
Mn6	9874(2)	2380(2)	2521(2)	26(1)	2.10
Mg1	137(2)	7609(2)	853(2)	24(1)	2.09
Mg2	-15(2)	4990(2)	1684(2)	27(1)	2.18
Mg3	0	1/2	1/2	27(1)	2.16
As1	8285(1)	9304(1)	3328(1)	28(1)	4.64
Si1	2842(3)	2735(2)	550(3)	23(1)	3.86
Si2	2838(3)	2735(3)	3620(3)	25(1)	3.87
Si3	7221(3)	4996(2)	3740(3)	22(1)	4.05
Si4	7254(3)	4995(2)	528(3)	22(1)	4.07
Si5	7100(3)	2606(2)	-2110(3)	21(1)	3.98
Si6	7041(3)	2587(2)	4788(3)	22(1)	4.01
O1	8665(7)	267(6)	2097(8)	28(2)	1.92
O2	9073(9)	8161(8)	2867(8)	34(3)	1.99
O3	6515(7)	8454(6)	3337(7)	24(2)	1.92
O4	1471(8)	-270(7)	4943(8)	30(3)	1.85
O5	3372(7)	3945(7)	-310(8)	29(3)	2.01
O6	-1172(7)	7903(6)	9509(8)	24(2)	1.99
O7	3604(7)	1647(6)	-85(7)	22(2)	1.89
O8	3258(7)	3509(6)	2279(8)	28(2)	1.95
O9	3609(7)	1649(6)	3234(8)	25(2)	1.84
O10	1177(7)	2121(6)	3777(8)	28(2)	1.91
O11	3410(7)	3955(7)	5112(8)	29(2)	2.04
O12	-3413(8)	-6453(7)	-5880(8)	31(3)	2.04
O13	8881(7)	5522(6)	3678(7)	23(2)	1.98
O14	-3351(7)	-5200(7)	-7847(7)	29(2)	2.08
O15	8924(7)	5499(6)	387(7)	24(2)	2.01
O16	6625(8)	3556(6)	-620(8)	30(2)	2.01
O17	8742(6)	2937(5)	-2155(7)	22(2)	1.94
O18	6171(6)	1037(5)	-2326(7)	19(2)	1.99
O19	-3274(7)	-6927(7)	-3429(8)	30(2)	2.03
O20	6098(6)	1025(5)	4265(6)	17(2)	1.89
O21	8684(7)	2896(6)	4524(7)	25(2)	2.00
OH1	1354(7)	9606(6)	1205(8)	29(2)	1.06
OH2	973(7)	4506(6)	2998(7)	24(2)	1.16
OH3	8845(7)	2918(6)	1158(7)	25(2)	1.13
OH4	5998(8)	996(6)	981(8)	27(2)	0.95

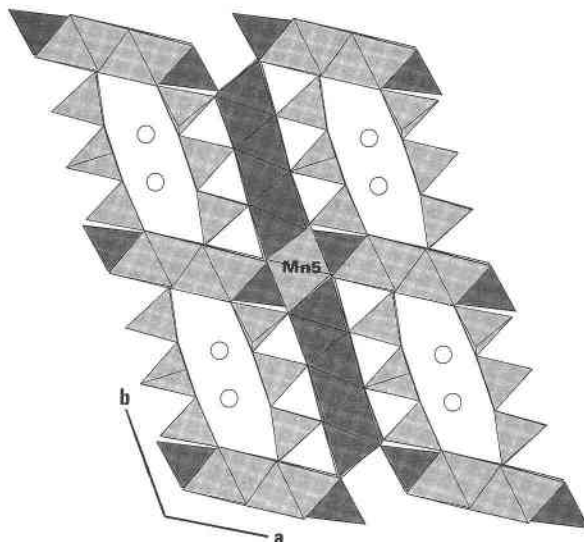


Fig. 1. A projection parallel to [001] of the johninnesite structure showing the layering parallel to {100}. Na atoms are represented by open circles, SiO_4 tetrahedra are lightly shaded, AsO_4 tetrahedra are darkly shaded, MnO_6 octahedra are lightly shaded, and $(\text{Mg,Mn})\text{O}_6$ octahedra are darkly shaded.

32%. Subsequent refinement, combined with difference maps, resulted in the location and correct scattering curve designation of the 42 atomic sites, with $R = 13.4\%$. Refining this model with 4078 observed reflections, $F_o > 6\sigma_F$, gave $R = 10.5$ and $R_w = 9.6\%$. Inspection of the list of structure factors revealed many reflections for which $|F_o| \gg |F_c|$, which the authors presumed to be the result of diffraction-peak overlap due to twinning. Dunn et al. (1986) noted a "twinning-like intergrowth," but the small size of their crystals prevented confirmation of this property. Oil-immersion mounts of several bladed crystal fragments under high magnification ($625\times$) with cross-polarized light showed twin lamellae parallel to [001]. Furthermore, close inspection of precession photographs revealed that johninnesite is twinned by 180° rotation around [001], with composition plane {010}. It was necessary to discard twin-overlap reflections with the largest ΔF values from a series of least-squares refinements, as was done in the structure refinement of ehrleite (Hawthorne and Grice, 1987). This process gradually caused the model to improve to $R = 7.5$ and $R_w = 6.3\%$ for the

3183 observed reflections (Table 1) and for all atoms refined with anisotropic displacement factors. Incorporation of an isotropic extinction parameter did not improve the R index, nor was there any significant change in R values when the crystal structure of johninnesite was refined in the noncentrosymmetric space group $P1$. Peak maxima and minima on the final residual difference map were approximately 2 and 1 e/\AA^3 respectively. The relatively high positive ΔF is a remnant of the twin overlap. Table 2 contains the final positional and equivalent isotropic displacement parameters, as well as refined occupancy parameters for the larger cations and bond valence sums for all sites. From the calculated bond valence sums (using the parameters of Brown, 1981), the correct valence may be assigned to each cation, and the number of OH^- anions determined to yield the simplest unit-cell formula, $\text{Na}_2\text{Mn}_3^{2+}(\text{Mg,Mn}^{2+})_7\text{As}_2^{5+}\text{Si}_{12}\text{O}_{42}(\text{OH})_8$. Interatomic distances are given in Table 3. Anisotropic displacement factors for all atoms (Table 4¹) and observed and calculated structure factors (Table 5¹) have been deposited.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The johninnesite structure consists of sheets parallel to {100} (Fig. 1). There are three distinct kinds of polyhedral sheet: (1) the tetrahedral layer, which consists of

¹ A listing of anisotropic displacement factors (Table 4) and observed and calculated structure factors (Table 5) may be ordered as Document AM-94-565 from the Business Office, Mineralogical Society of America, 1130 Seventeenth Street NW, Suite 330, Washington, DC 20036, U.S.A. Please remit \$5.00 in advance for the microfiche.

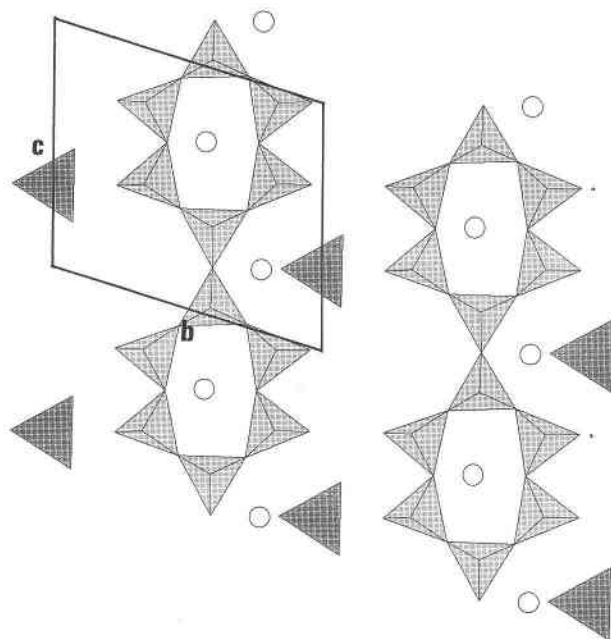


Fig. 2. The tetrahedral layer parallel to {100} in johninnesite, with independent AsO_4 tetrahedra and Si_6O_{17} loop-braided *vierer* single chains and Na atoms represented by open circles. The unit cell is outlined.

independent As tetrahedra and chains of Si tetrahedra parallel to the *c* axis in the configuration of a series of six-membered loops (Fig. 2); (2) the edge-sharing octahedral layer (Fig. 3), which consists of the larger Mn octahedra surrounding the holes in the sheet, whereas the somewhat smaller Mg and Mn octahedra serve as cross-linkages to complete the layer; and (3) another octahedral layer (Fig. 4) that consists of ribbons of edge-sharing Mn octahedra separated by independent, very distorted Na octahedra. The Na polyhedra actually resemble bifurcated tetragonal pyramids (Fig. 5a), or, if the two longer bond lengths of 3.2 Å are included, the coordination is distorted cubic (Fig. 5b). The structural formula of johninnesite may be written $\text{Na}_2\text{Mn}_9(\text{Mg}, \text{Mn})_7(\text{OH})_8[\text{AsO}_4]_2[\text{Si}_6\text{O}_{17}]_2$.

In Figure 1, the crystal structural basis for twinning in johninnesite may be seen. There is a pseudotwofold axis parallel to [001] through the central MnO_6 octahedron (the Mn5 site). Similarly, in Figures 2, 3, and 4, the {010} composition plane may be identified readily as a pseudomirror plane. Within the layering scheme, the edge-sharing octahedral layer (Fig. 3) is a modified brucite-like layer, with two of the 12 octahedral sites vacant ("holes"). Each of these paired holes is capped by one AsO_4 tetrahedron, one on top and the adjacent one on the bottom. The AsO_4 are part of the tetrahedral layer (T), which is sandwiched between the two distinctly different octahedral layers (O and O') described above. The layering sequence is . . . OTO'TOTO'TOTO' . . . , with the *a* unit-cell parameter defined by OTO'T.

Arsenosilicate minerals, with the exception of cervan-

TABLE 3. Selected interatomic distances (Å) for johninnesite

Na1-O3	2.404(7)	Mn1-O4	2.324(7)
Na1-O8	2.758(7)	Mn1-O9	2.091(9)
Na1-O5	2.637(10)	Mn1-O20	2.211(6)
Na1-O11	2.739(11)	Mn1-O3	2.198(6)
Na1-O14	2.402(11)	Mn1-O18	2.198(5)
Na1-O19	2.394(11)	Mn1-O20	2.361(7)
Na1-O12	3.254(10)	Mean	2.230
Na1-O16	3.187(12)		
Mn2-O1	2.522(8)	Mn3-O9	2.146(6)
Mn2-O3	2.192(9)	Mn3-OH4	2.315(8)
Mn2-O7	2.070(6)	Mn3-O7	2.138(8)
Mn2-O18	2.204(6)	Mn3-O18	2.232(8)
Mn2-O20	2.232(5)	Mn3-OH1	2.204(7)
Mn2-OH4	2.144(8)	Mn3-OH4	2.191(6)
Mean	2.227	Mean	2.204
Mn4-O2	2.115(10)	Mn5-O1	2.287(7)
Mn4-O4	2.110(6)	Mn5-O1	2.287(7)
Mn4-O10	2.130(7)	Mn5-O6	2.127(6)
Mn4-O13	2.237(6)	Mn5-O6	2.127(6)
Mn4-O17	2.292(6)	Mn5-OH1	2.197(9)
Mn4-O21	2.238(9)	Mn5-OH1	2.197(9)
Mean	2.187	Mean	2.204
Mn6-O21	2.182(7)	Mg1-OH1	2.053(6)
Mn6-OH3	2.151(9)	Mg1-O2	2.131(8)
Mn6-O1	2.151(6)	Mg1-O6	2.191(9)
Mn6-O6	2.220(7)	Mg1-O15	2.149(6)
Mn6-O10	2.102(9)	Mg1-O17	2.180(8)
Mn6-OH2	2.140(6)	Mg1-OH3	2.100(7)
Mean	2.158	Mean	2.134
Mg2-OH2	2.046(9)	Mg3-OH2	2.065(6)
Mg2-O13	2.133(7)	Mg3-O13	2.176(8)
Mg2-O15	2.105(9)	Mg3-O13	2.176(8)
Mg2-O15	2.172(7)	Mg3-O21	2.170(5)
Mg2-O17	2.132(5)	Mg3-O21	2.170(5)
Mg2-OH3	2.098(6)	Mg3-OH2	2.065(6)
Mean	2.114	Mean	2.137
Si1-O5	1.658(9)	Si2-O8	1.673(8)
Si1-O8	1.678(7)	Si2-O9	1.614(9)
Si1-O6	1.629(7)	Si2-O10	1.617(7)
Si1-O7	1.610(8)	Si2-O11	1.666(6)
Mean	1.644	Mean	1.642
Si3-O13	1.615(7)	Si4-O15	1.625(7)
Si3-O11	1.613(8)	Si4-O16	1.613(6)
Si3-O12	1.621(8)	Si4-O5	1.616(10)
Si3-O14	1.656(8)	Si4-O14	1.643(8)
Mean	1.626	Mean	1.624
Si5-O16	1.644(7)	Si6-O20	1.609(5)
Si5-O17	1.618(7)	Si6-O21	1.621(7)
Si5-O18	1.611(5)	Si6-O12	1.625(10)
Si5-O19	1.653(10)	Si6-O19	1.665(8)
Mean	1.632	Mean	1.630
As1-O1	1.729(8)		
As1-O2	1.678(10)		
As1-O3	1.740(6)		
As1-O4	1.694(7)		
Mean	1.710		

donite-(Ce) and asbecasite, are Mn-bearing. Such manganese arsenosilicates are relatively uncommon but have attracted the attention of metamorphic petrologists in recent years. Most of the 11 known manganese arsenosilicate species—ardennite, dixenite, hematolite, holdenite, kolicite, kraisslite, mcgovernite, nelenite, parwelite, schallerite, and tiragalloite—together with two Mn-free species (named above) occur infrequently and in small concentrations. Some quite rare species, among them asbecasite (Sacredoti et al., 1993), nelenite, whose structure is unstudied (Dunn and Peacor, 1984), schallerite (Kato and Watanabe, 1992), and cervandonite-(Ce) (Armbruster et al., 1988), contain trivalent As, which has a coor-

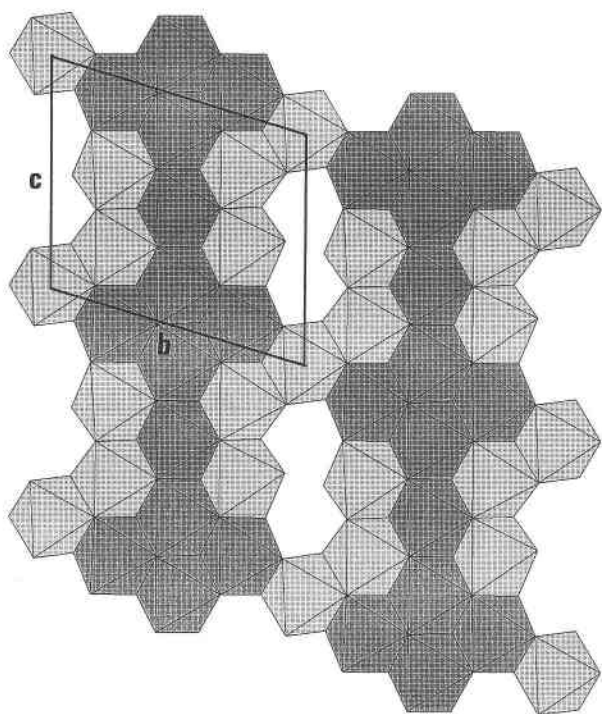


Fig. 3. The octahedral layer parallel to $\{100\}$ in johninnesite, with MnO_6 octahedra lightly shaded and $(\text{Mg,Mn})\text{O}_6$ darkly shaded. The unit cell is outlined.

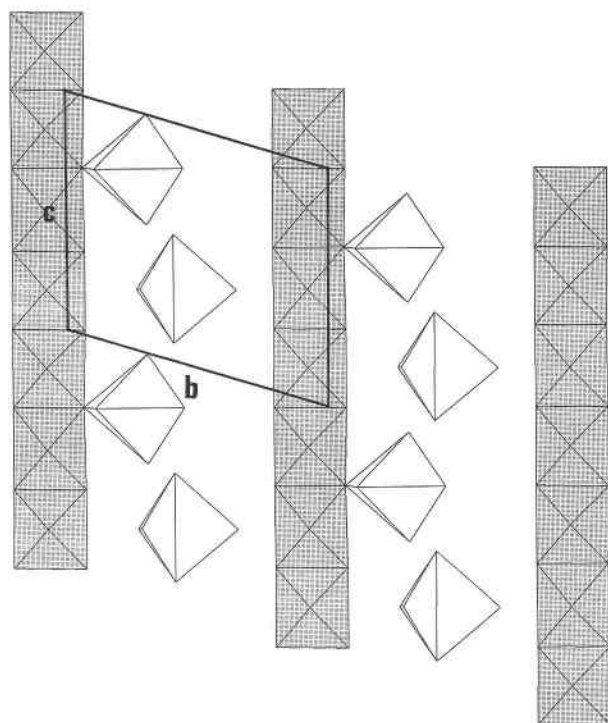


Fig. 4. A second octahedral layer parallel to $\{100\}$ in johninnesite showing ribbons of MnO_6 octahedra interspersed by very distorted NaO_6 polyhedra. The unit cell is outlined.

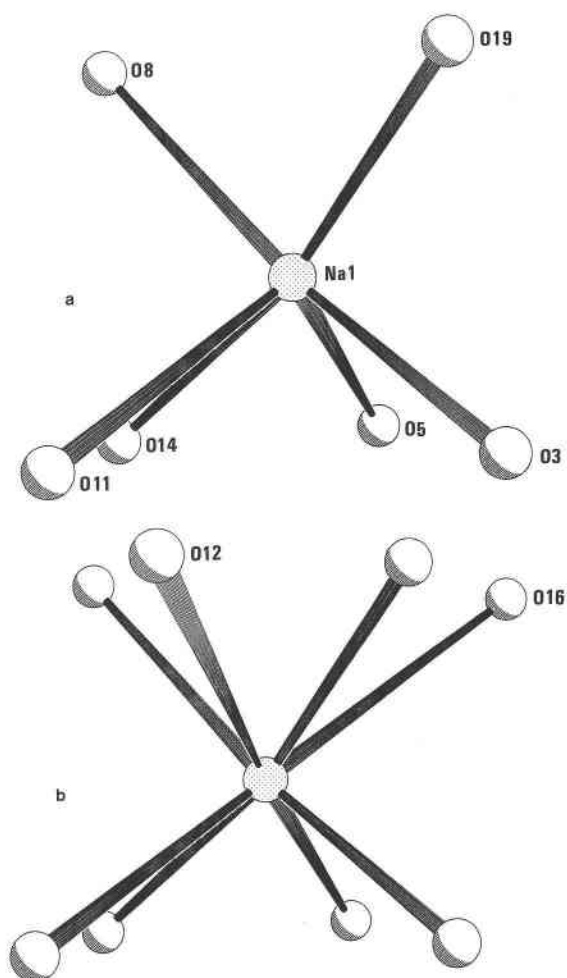


Fig. 5. The Na polyhedron in johninnesite (a) as a NaO_6 bifurcated tetragonal pyramid, and (b) as a NaO_6 distorted cube with the additional O atoms O12 and O16.

dination polyhedron described as a trigonal pyramid with a lone pair of electrons at the empty vertex.

Like the PO_4 and SiO_4 tetrahedra in silicophosphate minerals (Grice and McDonald, 1993), the tetrahedral cations in arsenosilicates (Si and As) are generally well ordered, and there is little to no polymerization of AsO_4 tetrahedra. The only structures of arsenosilicates that have distinct tetrahedral and octahedral layers as in johninnesite are asbecasite and schallerite. Asbecasite has a braided tetrahedral layer (Sacerdoti et al., 1993) that bears little resemblance to the topology of johninnesite, but schallerite (Kato and Watanabe, 1992), which has the structure of friedelite (Ozawa et al., 1983), incorporates some features similar to those found in johninnesite. Schallerite, with layering sequence . . .TOTO. . ., has a brucite-like layer of MnO_6 octahedra like johninnesite but without holes and an unbranched *sechser* single layer of tetrahedra (Liebau, 1985), in contrast to that described above for johninnesite.

Although johninnesite has crystal chemical and some topological similarities to other arsenosilicates, the fun-

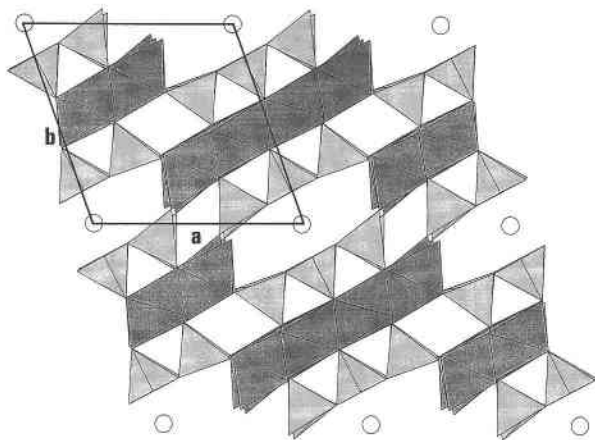


Fig. 6. A projection parallel to [001] of the howieite structure showing the layering parallel to {120}. The Si_6O_{17} chains constituting the tetrahedral layer are lightly shaded, and the $(\text{Mn,Mg,Fe})_6$ octahedra are darkly shaded. The Na atom is represented by open circles. The unit cell is outlined.

damental structural topology is different from any other member of this chemical class. To find a crystal structure with a topology comparable with that of johninnesite, we must look to the silicate class, since the dominant feature is the chains of Si tetrahedra. Liebau (1985) classified howieite, deerite, and taneyamalite as minerals with loop-branched, *vierer*, single-chain structures, similar to those found in johninnesite. Wenk (1974) described howieite, $\text{Na}(\text{Mn,Fe})_{12}[\text{Si}_6\text{O}_{17}]_2(\text{O,OH})_{10}$, as a layered silicate with a structure consisting of an octahedral layer, a tetrahedral layer, and Na atoms with eightfold coordination positioned between two tetrahedral layers (Fig. 6). The Si_6O_{17} chains of howieite and johninnesite are identical, but the fundamental differences in the tetrahedral layers of the two minerals are the incorporation of an arsenate group in johninnesite and that all tetrahedra point in the same direction in johninnesite, whereas in howieite adjacent chains have tetrahedra pointing in opposite directions. In howieite there is only one kind of octahedral layer, whereas there are two in johninnesite. Unlike howieite, the structure of deerite (Fleet, 1977), $(\text{Fe,Mn})_6(\text{Fe,Al})_3[\text{Si}_6\text{O}_{17}]\text{O}_3(\text{OH})_5$, is not layered. The looped chains of Si_6O_{17} tetrahedra and the cross-linking bands of octahedra do not have just one orientation but two, giving it a cross-hatched structural texture. The structure of taneyamalite has not been described, but it is considered to be the Mn^{2+} -dominant analogue of howieite.

From the discussion above, it is evident that the structure of johninnesite does not relate well to any other species in the arsenosilicate chemical class but does bear strong resemblances to the howieite structure type. The reason for this appears to be that, during crystallization

of the mineral, the AsO_4 tetrahedra play a passive role in the configuration of the structural topology, in contrast to the strong polymerization tendencies of silica.

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