

The crystal structure of nissonite

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ABSTRACT

The crystal structure of nissonite, $[\text{CuMg}(\text{PO}_4)(\text{OH})(\text{H}_2\text{O})_2]_2(\text{H}_2\text{O})$, monoclinic, $a = 22.523(5)$, $b = 5.015(2)$, $c = 10.506(3)\text{\AA}$, $\beta = 99.62(2)^\circ$, $V = 1169.9(6)\text{\AA}^3$, space group $C2/c$, $Z = 4$, has been solved by direct methods and refined to an R index of 6.8% for 1271 observed reflections ($>4\sigma$) measured on an automated four-circle single-crystal diffractometer using graphite-monochromated $\text{MoK}\alpha$ X-radiation. The structure consists of thick heteropolyhedral slabs parallel to (100), linked solely by a network of H bonds. Each slab consists of a sheet of corner-sharing $[\text{Cu}\phi_{10}]$ dimers sandwiched between two $[\text{M}(\text{T}\phi_4)\phi_3]$ sheets of corner-sharing ($\text{Mg}\phi_6$) and ($\text{P}\phi_4$) polyhedra; in each sheet, the polyhedra occupy the vertices of a 6^3 net. One of the H_2O groups in the formula unit is not bonded to Mg, Cu, or P but is held in the structure solely by a network of H bonds. Graphically identical $[\text{M}(\text{T}\phi_4)\phi_3]$ sheets occur in the structure of newberyite and as components of the heteropolyhedral frameworks of the structures of metavariscite and variscite.

INTRODUCTION

Nissonite is a hydrous copper magnesium phosphate mineral described by Mrose et al. (1966). It occurs as thin crusts and occasional diamond-shaped crystals associated with such other Cu minerals as malachite, azurite, libethenite, turquoise, chrysocolla, and cuprite. The only other data in the literature are reported by Bayliss (1986), who gave improved X-ray powder data and cell dimensions; he also noted the paucity of material and derived the data by remeasuring the original Debye-Scherrer film of nissonite recorded by Mrose et al. (1966).

We have been investigating the graphical and topological aspects of the Cu^{2+} oxysalts for some time (Hawthorne, 1985a, 1985b, 1986; Hawthorne and Groat, 1985, 1986; Hawthorne and Eby, 1985; Hawthorne et al., 1989; Groat and Hawthorne, 1987; Eby and Hawthorne, 1989a, 1989b), and the structure of nissonite was of obvious interest.

EXPERIMENTAL

The material used in this work is from Panoche Valley, California, the original locality from which the mineral was described by Mrose et al. (1966) and the only locality from which it has been reported; the sample was obtained from a mineral dealer. As described by Bayliss (1986), the material available is extremely limited, and a considerable amount of time was spent examining unsuitable crystals. Finally, a crystal was found that gave only slightly diffuse spots, and this was used for the measurement of the metric and intensity data.

The crystal was mounted on a Nicolet R3m automated four-circle diffractometer, and a random orientation photograph was used to locate 15 reflections that were automatically centered using graphite-monochromated $\text{MoK}\alpha$ X-radiation. The correct unit cell was chosen from an array of real space vectors corresponding to potential unit-cell axes, and least-squares refinement of the setting angles resulted in the cell parameters reported in Table 1. Intensity data were measured according to the method of Hawthorne and Groat (1985). A total of 1702 reflections was measured out to a maximum 2θ value of 60° . Two standard reflections were monitored every 48 reflections to check for stability and constancy of crystal alignment; no significant change was noted during data measurement. The intensity data were corrected for absorption, Lorentz, and polarization effects, and reduced to structure factors. A reflection was considered as observed if its magnitude exceeded that of three standard deviations based on counting statistics; of the 1702 unique reflections, 1271 were considered as observed.

STRUCTURE SOLUTION AND REFINEMENT

Scattering curves for neutral atoms together with anomalous dispersion corrections were taken from the *International Tables for X-ray Crystallography* (1974); R indices are of the form given in Table 1 and are expressed as percentages. All calculations were done with the SHELXTL system of programs (Sheldrick, 1981).

The structure was determined using the weighted tangent formula method. The phase set with the maximum combined figure of merit resulted in an E-map, the most intense peaks of which could be interpreted in terms of the known ratios (and intensities) of the cations in nissonite. Most of the O atoms were located on the subsequent difference-Fourier map. Iterative full-matrix least-squares

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TABLE 1. Miscellaneous data for nissonite

<i>a</i>	22.523(5) Å	Crystal size (mm)	0.10 × 0.16 × 0.16
<i>b</i>	5.015(2)	Rad/mono	Mo/Gr
<i>c</i>	10.506(3)	Total $ F_o $	1702
β	99.62(2) ^a	$ F_o > 4\sigma$	1271
<i>V</i>	1169.9(6) Å ³	Final $R_{w,obs}$	6.8%
Space group	C2/c	Final $R_{w,obs}$	6.0%
Unit-cell contents: 4{[CuMg(PO ₄)(OH)(H ₂ O) ₂](H ₂ O)}			
$R = \sum (F_o - F_c) / \sum F_o $			
$R_w = [\sum w(F_o - F_c)^2 / \sum wF_o^2]^{1/4}$, $w = 1$			

refinement and difference-Fourier maps located the remaining non-H atoms, eventually converging to an *R* index of 7.1% for an isotropic displacement model. A bond-valence calculation identified the O²⁻, OH, and H₂O anions, and these were relabeled accordingly. Displacement factors were converted to anisotropic parameters, and the structure was refined to convergence. A difference-Fourier map calculated at this stage did not show any H-atom positions that are structurally reasonable. Consequently, a H-bond network was deduced on the basis of bond-valence and geometrical criteria, and H positions were derived; these were put into the structure model as fixed parameters. Subsequent full-matrix least-squares refinement of all variables resulted in convergence at an *R* index of 6.0%; this value is higher than one would normally expect and reflects the rather inferior nature of the material. Final atomic parameters are given in Tables 2 and 3, and observed and calculated structure factors are deposited (Table 4)¹. Selected interatomic distances and angles are given in Table 5, and an empirical bond-valence table, calculated with the curves of Brown (1981), is shown as Table 6.

¹ To receive a copy of Table 4, order Document Am-90-439 from the Business Office, Mineralogical Society of America, 1130 Seventeenth Street NW, Suite 330, Washington, DC 20036. Please remit \$5.00 in advance for the microfiche.

TABLE 2. Atomic parameters for nissonite

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*]
Cu	0.24534(5)	0.1541(3)	0.36677(9)	0.96(3)
P	0.3437(1)	0.1768(6)	0.2264(2)	0.83(6)
Mg	0.1226(1)	0.1547(8)	0.4673(2)	0.73(8)
O(1)	0.3631(3)	0.103(1)	0.3702(5)	1.4(2)
O(2)	0.2770(3)	0.296(1)	0.2161(5)	0.9(2)
O(3)	0.3846(3)	0.388(1)	0.1833(5)	1.2(2)
O(4)	0.3405(3)	-0.072(2)	0.1447(5)	1.3(2)
OH	0.2136(3)	0.055(1)	0.5211(5)	0.7(1)
OW(1)	0.0866(3)	-0.138(2)	0.5790(5)	1.5(2)
OW(2)	0.0359(3)	0.288(2)	0.4132(6)	2.4(3)
OW(3)	1/2	0.201(3)	1/4	2.8(4)
H(OH)	0.245	0	0.578	1.5
H(W1a)	0.105	0	0.598	1.5
H(W1b)	0.057	0.015	0.639	1.5
H(W2a)	0.023	0.434	0.355	1.5
H(W2b)	0	0.237	0.415	1.5
H(W3)	0.460	0.268	0.227	1.5

* $U_{iso} = U_{eq} \times 10^3$; H positions derived from bond-valence and stereochemical arguments.

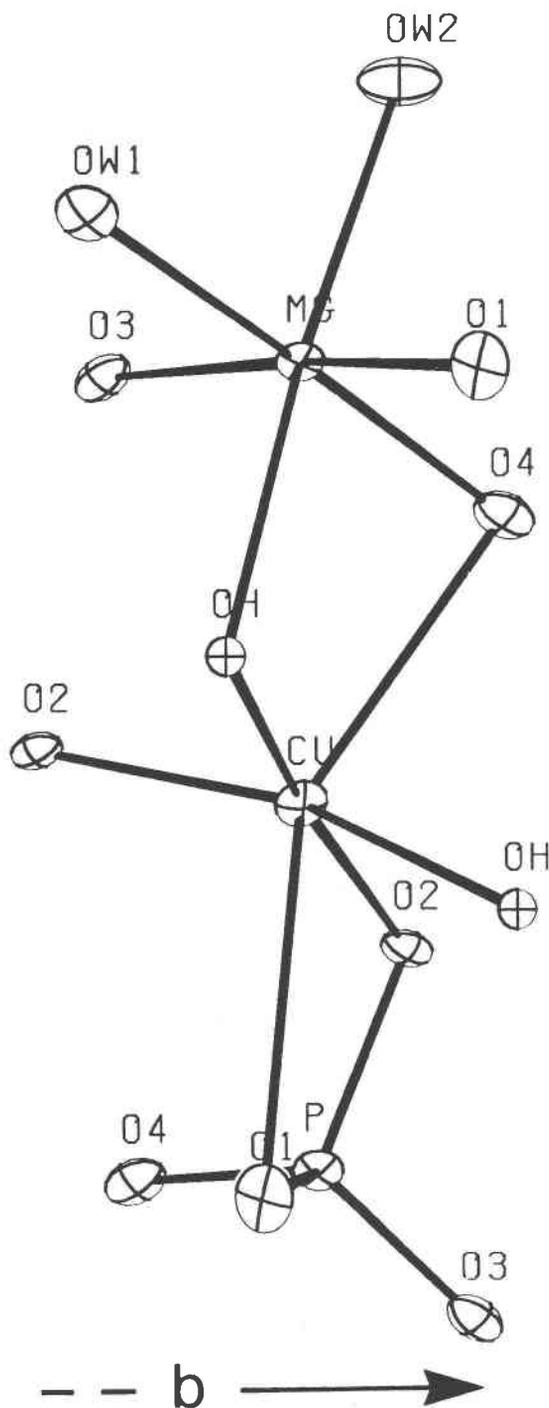


Fig. 1. Details of the local polyhedral geometry in nissonite projected along [001].

STRUCTURE DESCRIPTION

There is one unique P position in nissonite, tetrahedrally coordinated by four O atoms; the resulting oxyanion shows a typical range of bond lengths and angles (cf. Baur, 1974). The variation in P-O bond lengths cor-

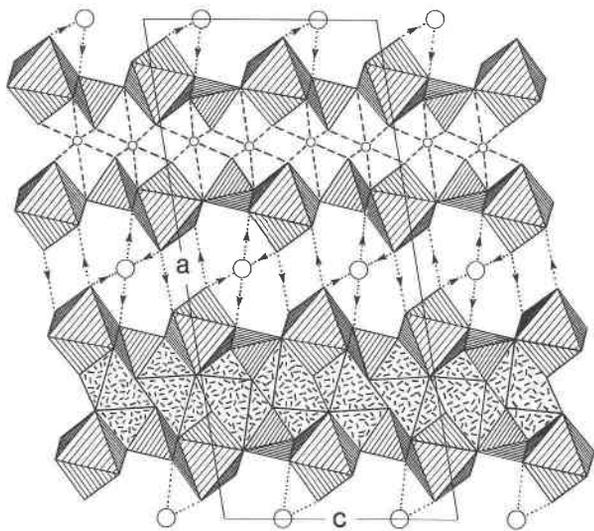


Fig. 2. Polyhedral representation of the structure of nissonite projected down [010]. (Mgφ₆) and (Pφ₄) polyhedra are shown ruled, and (Cuφ₆) polyhedra are shown by dashed shading; the small circles in the upper part of the figure represent Cu atoms, and Cu-O bonds are indicated by the broken lines; the large circles represent the H₂O groups that are not directly bonded to Mg, Cu, or P, and the dotted lines represent H bonds with the donor-acceptor character indicated by the arrows. One unit cell is outlined.

relates well with the local bond-valence requirements of the corresponding O anions. There is one Mg position, coordinated by six anions in a (distorted) octahedral arrangement; of these, three are (phosphate) O atoms, one is an OH, and the remaining two are H₂O groups, the latter being in a *cis* relationship. There is one unique Cu²⁺ position, surrounded by six anions in a very distorted octahedral arrangement, the Cu-φ distances varying from 1.94–2.66 Å. This type of geometry is typical for Cu²⁺, being a result of the Jahn-Teller effect. Details of the local geometry are shown in Figure 1; note that the three unique polyhedra are connected by shared edges, all of which are shortened relative to the unshared edges of the same polyhedra.

TABLE 3. Anisotropic displacement factor coefficients for nissonite

Atom	U ₁₁ ^a	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
Cu	86(5)	131(6)	55(4)	-16(5)	-33(3)	-23(6)
P	61(10)	115(13)	56(8)	2(10)	-43(7)	13(12)
Mg	49(13)	108(16)	44(11)	-1(13)	-40(10)	-10(16)
O(1)	177(34)	167(43)	59(24)	13(25)	-63(23)	59(32)
O(2)	58(28)	123(40)	88(25)	5(25)	-23(21)	34(29)
O(3)	83(30)	129(41)	122(26)	35(26)	-19(23)	-5(30)
O(4)	85(32)	169(39)	123(28)	-36(26)	-38(24)	8(31)
OH	71(12)					
OW(1)	127(31)	176(39)	131(27)	-8(31)	-47(23)	-47(36)
OW(2)	59(32)	355(56)	263(36)	0	-55(27)	0
OW(3)	90(47)	292(76)	388(57)	0	-134(42)	0

^a U_y = U_y × 10³.

TABLE 5. Interatomic distances (Å) and angles (°) in nissonite

Cu-O(1)	2.660(7)	Mg-OH	2.095(7)
Cu-O(2)	1.975(6)	Mg-OW(1)	2.122(8)
Cu-OH	1.943(6)	Mg-OW(2)	2.051(7)
Cu-O(2)a	2.021(7)	Mg-O(1)c	2.075(7)
Cu-O(4)b	2.360(7)	Mg-O(3)a	2.057(7)
Cu-OHc	2.003(6)	Mg-O(4)b	2.071(8)
(Cu-O)	2.160	(Mg-O)	2.079
P-O(1)	1.545(6)		
P-O(2)	1.604(7)		
P-O(3)	1.523(7)		
P-O(4)	1.507(7)		
(P-O)	1.545		
O(1)-O(2)*	2.504(8)	O(1)-Cu-O(2)*	63.4(2)
O(1)-OH	3.963(9)	O(1)-Cu-OH	118.0(2)
O(1)-O(2)a	3.493(9)	O(1)-Cu-O(2)a	95.6(2)
O(1)-OHc	2.81(1)	O(1)-Cu-OHc	72.6(2)
O(2)-O(2)a	2.927(7)	O(2)-Cu-O(2)a	94.2(2)
O(2)-O(4)b	3.299(9)	O(2)-Cu-O(4)b	98.7(2)
O(2)-OHc	2.834(8)	O(2)-Cu-OHc	90.8(3)
OH-O(2)a	2.847(8)	OH-Cu-O(2)a	91.8(3)
OH-O(4)b*	2.707(9)	OH-Cu-O(4)b*	77.3(2)
OH-OHc*	2.64(1)	OH-Cu-OHc*	83.9(3)
O(2)a-O(4)b	3.608(9)	O(2)a-Cu-O(4)b	110.6(2)
O(4)b-OHc	2.937(9)	O(4)b-Cu-OHc	84.2(2)
(O-O Cu)	3.05	(O-Cu-O)	90.1
OH-OW(1)	3.173(9)	OH-Mg-OW(1)	97.6(3)
OH-O(1)c	2.81(1)	OH-Mg-O(1)c	84.7(3)
OH-O(3)a	2.935(8)	OH-Mg-O(3)a	90.0(3)
OH-O(4)b*	2.707(9)	OH-Mg-O(4)b*	81.1(3)
OW(1)-OW(2)	2.87(1)	OW(1)-Mg-OW(2)	86.8(3)
OW(1)-O(1)c	2.92(1)	OW(1)-Mg-O(1)c	88.3(3)
OW(1)-O(3)a	2.938(8)	OW(1)-Mg-O(3)a	89.3(3)
OW(2)-O(1)c	2.986(8)	OW(2)-Mg-O(1)c	92.7(3)
OW(2)-O(3)a	2.98(1)	OW(2)-Mg-O(3)a	92.8(3)
OW(2)-O(4)b	3.03(1)	OW(2)-Mg-O(4)b	94.6(3)
O(1)c-O(4)b	3.015(9)	O(1)c-Mg-O(4)b	93.3(3)
O(3)a-O(4)b	2.890(9)	O(3)a-Mg-O(4)b	88.9(3)
(O-O Mg)	2.94	(O-Mg-O)	90.0
O(1)-O(2)*	2.504(8)	O(1)-P-O(2)*	105.3(4)
O(1)-O(3)	2.538(9)	O(1)-P-O(3)	111.7(4)
O(1)-O(4)	2.497(8)	O(1)-P-O(4)	109.8(4)
O(2)-O(3)	2.546(9)	O(2)-P-O(3)	109.0(4)
O(2)-O(4)	2.52(1)	O(2)-P-O(4)	108.4(4)
O(3)-O(4)	2.52(1)	O(3)-P-O(4)	112.3(4)
(O-O P)	2.52	(O-P-O)	109.4
OH-H(OH)	0.89	OW(2)-H(W2a)	0.97
OH-O(2)d	2.89	OW(2)-H(W2b)	0.85
OH-O(4)d	2.94	OW(2)-OW(3)f	2.73
H(OH)-O(2)d	2.12	OW(2)-OW(1)e	2.87
H(OH)-O(4)d	2.18	H(W2a)-OW(3)f	1.76
		H(W2b)-OW(1)e	2.02
OW(1)-H(W1a)	0.81		
OW(1)-H(W1b)	1.25	OW(3)-H(W3)	0.95
OW(1)-O(1)c	2.93	OW(3)-O(3)g	2.74
OW(1)-O(3)c	2.77	H(W3)-O(3)g	1.79
H(W1a)-O(1)c	2.12		
H(W1b)-O(3)c	2.15		

Note: a: 1/2 - x, y - 1/2, 1/2 - z; b: 1/2 - x, 1/2 + y, 1/2 - z; c: 1/2 - x, 1/2 - y, 1 - z; d: x, -y, 1/2 + z; e: -x, -y, 1 - z; f: -1/2 + x, 1/2 + y, 1/2 - z; g: 1 - x, y, 1/2 - z.

* Shared edge.

Figure 2 shows the structure projected down [010]. In this orientation, the structure exhibits a very layered character. The (Cuφ₆) (φ = unspecified ligand), (Mgφ₆) and (Pφ₄) polyhedra form thick slabs parallel to (100). Intercalated between these slabs are planar layers of (H₂O)

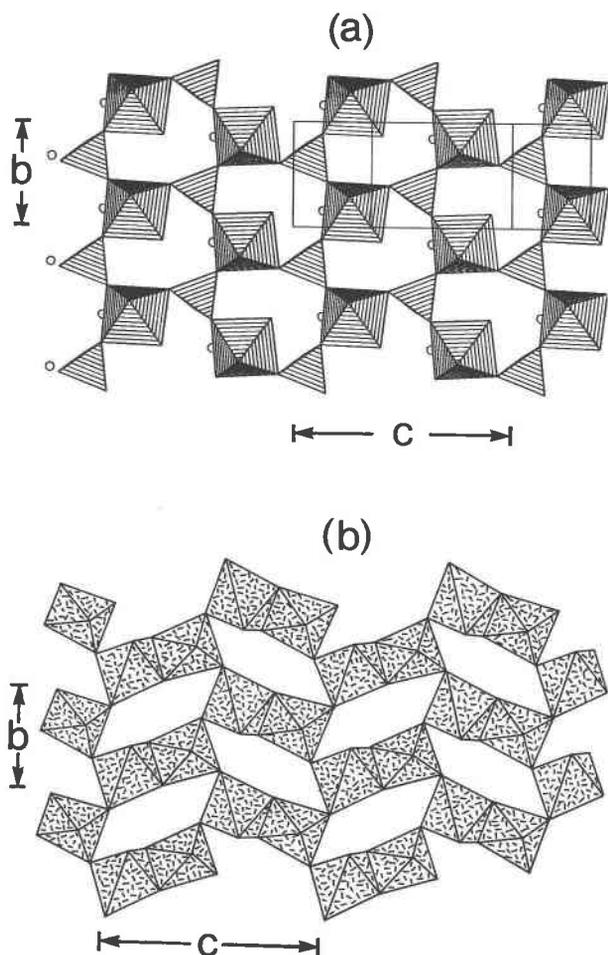


Fig. 3. The sheet elements of the nissonite structure viewed down [100]; the legend is as in Figure 2. (a) The $[\text{Mg}(\text{PO}_4)(\text{OH})(\text{H}_2\text{O})_2]$ sheet, consisting of alternating $(\text{Mg}\phi_6)$ and $(\text{P}\phi_4)$ polyhedra arranged at the vertices of a 6^3 net and (b) the $[\text{Cu}\phi_6]$ sheet, consisting of edge-sharing dimers of $(\text{Cu}\phi_6)$ octahedra that share corners to form an array with the polyhedra at the vertices of a 6^3 net.

groups that provide the necessary interslab linkage by a H-bond network; this overall structure accounts nicely for the $\{100\}$ cleavage shown by nissonite. As is apparent

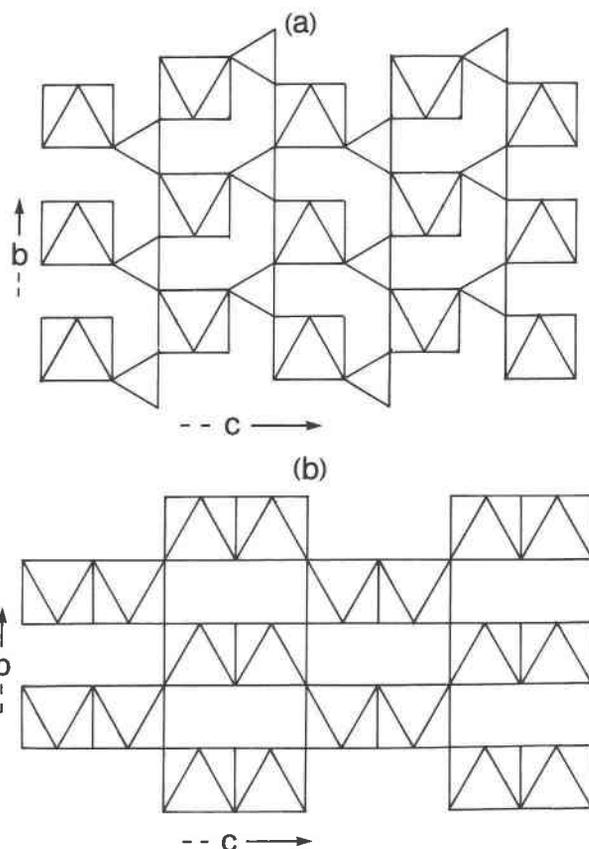


Fig. 4. Geometrically idealized versions of the polyhedral sheets shown in Figure 3. (a) The $[\text{M}(\text{T}\phi_6)\phi_6]$ sheet and (b) the $[\text{Cu}\phi_6]$ sheet.

from Figure 2, each slab consists of three polyhedral sheets, a central sheet of $(\text{Cu}\phi_6)$ octahedra sandwiched between heteropolyhedral sheets of $(\text{Mg}\phi_6)$ octahedra and $(\text{P}\phi_4)$ tetrahedra.

The constitution of the sheets is illustrated in Figure 3. The sandwiching Mg-P nets are symmetrically equivalent; one is illustrated in Figure 3a. It consists of a corner-sharing array of $(\text{Mg}\phi_6)$ and $(\text{P}\phi_4)$ polyhedra with unit stoichiometry $[\text{Mg}(\text{PO}_4)(\text{OH})(\text{H}_2\text{O})_2]$. In this orientation,

TABLE 6. Empirical bond-valence* table for nissonite

	Cu	Mg	P	H(1)	H(2)	H(3)	H(4)	H(5)	H(6)	Σ
O(1)	0.072	0.345	1.232		0.13					[1.779]
O(2)	0.376		1.046	0.15						[2.004]
	0.432									
O(3)		0.359	1.321			0.19			0.19	[2.060]
O(4)	0.149	0.348	1.379	0.12						[1.996]
OH	0.476	0.328		0.73						[1.933]
	0.399									
OW(1)		0.323			0.87	0.81		0.16		[2.163]
OW(2)		0.355					0.80	0.84		[1.995]
OW(3)							0.20 ^{±2}		0.81 ^{±2}	[2.020]
Σ	1.904	2.058	4.978	1.0	1.0	1.0	1.0	1.0	1.0	

* Calculated from the curves of Brown (1981).

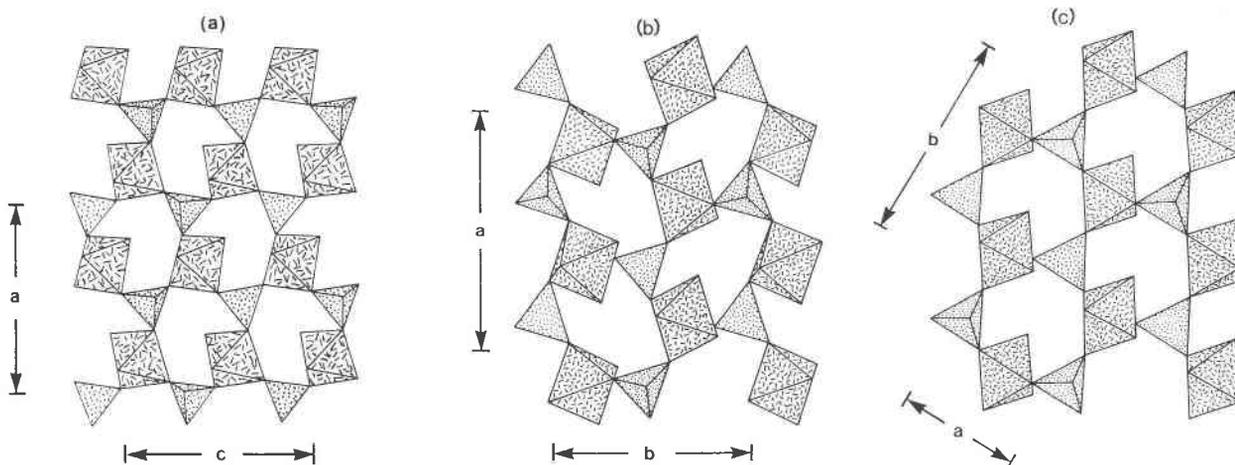


Fig. 5. The structural units in (a) newberyite (Sutor, 1967); (b) metavariscite (Kniep and Mootz, 1973); and (c) variscite (Kniep et al., 1977).

the sheet links outward (along *a*) to the $[\text{Cu}\phi_n]$ sheet. It is notable that the connectivity of the polyhedra is that of a 6^3 net. A geometrically idealized version of this sheet is shown in Figure 4a.

The central $[\text{Cu}\phi_n]$ sheet consists of edge-sharing $[\text{Cu}_2\text{O}_8(\text{OH})_2]$ dimers that link by corner-sharing [through O(2) atoms] to form the sheet shown in Figure 3b. This sheet may be geometrically idealized to that shown in Figure 4b, where the polyhedral connectivity is based on that of a 6^3 net. The relationship between the actual sheet and its idealized analogue is immediately apparent by comparing Figures 3b and 4b; the observed sheet has a commensurate modulation along *c*. The origin of this modulation stems from the requirement that this sheet link to the sandwiching $[\text{Mg}(\text{P}\phi_4)\phi_3]$ sheets. In the idealized $[\text{Cu}\phi_n]$ sheet, the anions that link to the neighboring $[\text{Mg}(\text{P}\phi_4)\phi_3]$ sheet form a (geometrically distorted) 6^3 net. The modulation brings this array of anions much closer to a geometrically ideal 6^3 net, matching it with the corresponding array in the adjacent $[\text{Mg}(\text{P}\phi_4)\phi_3]$ net.

COMPARISON WITH RELATED STRUCTURES

As discussed above, the strongly-bonded structural unit in nissonite consists of a sheet of $(\text{Cu}\phi_6)$ octahedra, sandwiched between heteropolyhedral $[\text{Mg}(\text{PO}_4)(\text{OH})(\text{H}_2\text{O})_2]$ sheets; the latter may be written in the general form $[\text{M}(\text{T}\phi_4)\phi_3]$, where M = six-coordinated cations, T = four-coordinated cations, ϕ = unspecified anions. Graphically identical $[\text{M}(\text{T}\phi_4)\phi_3]$ sheets are found in several other minerals. A similar sheet constitutes the structural unit in newberyite, $[\text{Mg}(\text{PO}_3\text{OH})(\text{H}_2\text{O})_3]$; this is illustrated in Figure 5a, and a comparison with the sheet of Figure 3a shows a subtle difference between the two sheets. In nissonite, each tetrahedron has one face in the plane of the sheet, and all tetrahedra point in the same direction, toward the central $[\text{Cu}\phi_n]$ sheet. In newberyite, each tetrahedron has one face in the plane of the sheet, but alternate tetrahedra point different ways (along *b*). Consequently,

the sheets in nissonite and newberyite are graphically identical but are geometrical isomers (Hawthorne, 1983, 1985c).

Graphically identical sheets are also found in the structures of metavariscite, $[\text{Al}(\text{PO}_4)(\text{H}_2\text{O})_2]$, and variscite, $[\text{Al}(\text{PO}_4)(\text{H}_2\text{O})_2]$ (Figs. 5a and 5b); in both of these structures, adjacent $[\text{M}(\text{T}\phi_4)\phi_3]$ sheets condense to form heteropolyhedral frameworks. Both sheets have the same tetrahedral conformation as the heteropolyhedral sheet in nissonite, with alternate tetrahedra pointing in opposite directions orthogonal to the plane of the sheet. However, there are still geometrical differences between the sheets. In metavariscite and variscite, three tetrahedra link to each octahedron such that the common vertices are all *cis* to each other (Figs. 5b and 5c). In nissonite, the tetrahedra link to each octahedron such that the linking vertices are both *cis* and *trans*. Consequently, the sheets in nissonite and in metavariscite and variscite are also geometrical isomers.

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