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Minyulite: its atomic arrangement

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Abstract

Minyulite, $K[Al_2F(H_2O)_4(PO_4)_2]$, orthorhombic, space group *Pba*2, *a* 9.337(5), *b* 9.740(5), *c* 5.522(3)A, $Z = 2$, possesses rigid dimeric clusters of formula $[Al_2F(H_2O)_4(PO_4)_2(O_P)_2]$, topologically identical to the $[Fe_2^{3+}(OH)(H_2O)_4(SO_4)_2(O_S)_2]$ clusters in copiapite, where O_P and O_S correspond to the phosphate and sulfate ligands, respectively. The structure was solved by Patterson and Fourier syntheses. Hydrogen atoms were located by difference synthesis. Least-squares refinement converged to $R(hkl) = 0.022$ for 618 reflections.

The dimeric cluster consists of two Al^{3+}–O octahedra sharing one F^- vertex and two PO_4 tetrahedra, which further link the octahedra. The clusters link via shared octahedral–tetrahedral vertices to form sheets parallel to {001}. Cavities in the sheets are occupied by K^+ ions. One tetrahedral vertex is unshared and accepts four hydrogen bonds from water molecules coordinated to Al^{3+}. These hydrogen bonds provide the only linkage between the sheets.

The greater affinity of Al^{3+} for F^- than $(OH)^-$ and the violation of electrostatic valence balance when F^- bonds to more than two Al^{3+} cations are suggested as the reasons for the condensation of Al^{3+} octahedra into dimers and chains in aqueous solution, leading ultimately to the crystallization of minyulite and fluellite. The leucophosphite structure may be preferred in the absence of F^-.

Introduction

Minyulite was originally described by Simpson and LeMesurier (1933) as a new species from Minyulo Well, Western Australia, where it occurs as radiating groups of fine fibers on the surfaces of crevices in phosphatic ironstone. Using optical methods, they established orthorhombic symmetry and proposed the formula $KAl_2(OH,F)(PO_4)_2 \cdot 3.5H_2O$. Spencer *et al.* (1943) studied minyulite from Wait's quarry near Noarlunga, South Australia, where it occurs in phosphate rock as short prismatic crystals in sub-parallel growth coating apatite. From X-ray oscillation photographs, they determined the cell constants *a* 9.35, *b* 9.74, *c* 5.52A and the space group *Pmm*2, the hemimorphic nature of the crystals being established by etch figures. The formula $KAl_2(PO_4)_2(OH,F) \cdot 4H_2O$, $Z = 2$ was suggested.

Haseman *et al.* (1950) synthesized minyulite, as well as a number of other hydrous K^+–NH_4^+, Al^{3+}–Fe^{3+}, phosphates, including leucophosphite, $KFe_2^{3+}(OH)(PO_4)_2 \cdot 2H_2O$, and its Al^{3+} analog. They noted that minyulite and leucophosphite can be synthesized by the treatment of clays with phosphate anions at pH ranges appropriate for soil environments and at temperatures less than 95°C. They proposed that these minerals may be important in the fixation of inorganic phosphate anions in soils.

Leucophosphite bears a striking chemical similarity to minyulite, and besides their possible formation under similar conditions in the soil, the report of leucophosphite in phosphatic rock associated with iron ore at Bomi Hill and Bambuta, Liberia, by Axelrod *et al.* (1952) is further indication of similar parageneses for these two minerals. A comparison of the structures of minyulite and leucophosphite may lead to an understanding of the conditions necessary for their formation.

Also of interest for structural comparison is the mineral fluellite, $Al_2F_2(OH)(H_2O)_3(PO_4) \cdot 4H_2O$. Murray (1973) reported fluellite occurring associated with minyulite in phosphate rock near Wolffdene, Queensland, Australia, and fluellite has been verified

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on a specimen of phosphate rock from Moculta Quarry, Angaston, South Australia, provided by J. E. Johnson of the South Australian Museum.

Experimental

The crystal studied was extracted from a specimen from Wait's Quarry, kindly provided by J. E. Johnson. Some difficulty was encountered in finding a suitable single crystal, owing to the sub-parallel growth of individuals. By splitting crystal clusters, an acceptable crystal fragment was obtained, measuring 0.07 mm along the *a* and *b* axes, and 0.10 mm along the *c* axis. The crystal was mounted parallel to the *c* axis.

Precession photographs indicated systematic absences corresponding to the space groups *Pbam* and *Pba*2. The intensities of 1315 reflections (maximum $2\theta = 55°$) were gathered on a Picker automated diffractometer utilizing graphite-monochromatized Mo*Kα* radiation. Very small mosaic spread permitted narrow half-angle scans of 0.4° with a scan rate of 1.0°/minute. Twenty-second background counting times were used on either side of each reflection. Least-squares refinement of 30 reference reflections yielded the cell constants given in Table 1.

Systematic absences indicated by the intensity data confirmed the space group *Pbam* or *Pba*2. The data were processed by conventional computational procedures to obtain $|F(\text{obs})|$. No absorption correction was applied, owing to the favorable size and shape of the crystal and the small absorption coefficient ($\mu = 11.0\ \text{cm}^{-1}$). After elimination of the extinct reflections and averaging of the symmetry equivalent reflections, 618 independent reflections remained for the ensuing study.

Solution and refinement of the structure

Three-dimensional Patterson synthesis, *P*(*uvw*), revealed the positions of the K, Al, and P atoms and determined the space group to be *Pba*2. Subsequent Fourier synthesis resolved the positions of all the remaining non-hydrogen atoms.

TABLE 1. Minyulite. Crystal cell parameters

a(Å)	9.337(5)
b(Å)	9.740(5)
c(Å)	5.522(3)
Space group	Pba2
Z	2
Formula	$KAl_2F(H_2O)_2(PO_4)_2$
Specific gravity[1]	2.46
Density (calc) gm/cm^3	2.47

[1] Spencer *et al.* (1943).

TABLE 2. Minyulite. Atomic coordinate parameters

Atom	Mult.	x	y	z
K	2	0.0000	0.0000	0.0000
Al	4	0.3591(1)	0.1105(1)	0.9545(4)
P	4	0.1470(1)	0.3243(1)	0.2040(4)
O(1)	4	0.4832(2)	0.1789(3)	0.1798(5)
O(2)	4	0.2075(3)	0.4658(3)	0.1382(6)
O(3)	4	0.2075(3)	0.2168(2)	0.0310(6)
O(4)	4	0.1850(2)	0.2918(3)	0.4675(6)
OW(1)	4	0.2383(3)	0.0429(3)	0.6793(7)
OW(2)	4	0.4285(3)	0.2511(3)	0.7202(7)
F	2	0.0000	0.5000	0.8217(6)
H(1)	4	0.226(4)	0.110(4)	0.596(8)
H(2)	4	0.234(4)	0.486(5)	0.597(9)
H(3)	4	0.352(4)	0.263(4)	0.606(8)
H(4)	4	0.002(4)	0.281(4)	0.608(8)

The atomic coordinates and thermal vibration parameters were refined using the NUCLS program, a modified version of ORFLS by Busing *et al.* (1962). Three cycles of full-matrix atomic parameter and isotropic thermal vibration parameter refinement converged to $R(hkl) = 0.044$. Two cycles of refinement using anisotropic thermal vibration parameters converged to $R(hkl) = 0.033$. A difference Fourier synthesis yielded the locations of four unique H atoms. A final two cycles of refinement, including hydrogen atoms and applying a secondary extinction correction, converged to $R = 0.022$ ($Rw = 0.024$). The extinction parameter converged to $4.0(2) \times 10^{-6}$.

Initially an oxygen atom was assigned to the atomic position (0 ½ 0.82). This yielded a negative temperature factor. During the final cycles of refinement, fluorine was assigned to this location, and the final thermal vibration parameters were comparable to those of the other anions. A fluorine determination by Spencer *et al.* (1943) yielded 2.7 weight percent F, indicating at least two fluorine atoms per unit cell.

Atomic scattering factors for K°, Al°, P°, O°, and F° were obtained from Cromer and Mann (1968) and included the anomalous dispersion terms for K, Al, P, and O. Scattering factors for H were obtained from Stewart *et al.* (1965). Final atomic coordinates are presented in Table 2 and anisotropic thermal

TABLE 3. Minyulite. Anisotropic thermal vibration parameters*

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K	0.0031(1)	0.0049(1)	0.0172(6)	-0.0007(1)		
Al	0.0016(1)	0.0016(1)	0.0055(4)	0.0001(1)	-0.0003(2)	-0.0001(2)
P	0.0016(1)	0.0016(1)	0.0047(3)	0.0023(1)	0.0006(2)	0.0006(2)
O(1)	0.0016(2)	0.0029(2)	0.0070(9)	-0.0001(2)	0.0001(5)	-0.0006(5)
O(2)	0.0020(3)	0.0018(2)	0.0119(11)	0.0001(2)	-0.0009(4)	0.0007(5)
O(3)	0.0023(3)	0.0023(2)	0.0089(10)	0.0007(2)	0.0000(4)	-0.0013(5)
O(4)	0.0028(3)	0.0037(3)	0.0070(9)	0.0003(2)	-0.0004(5)	0.0009(5)
OW(1)	0.0035(3)	0.0029(3)	0.0071(10)	0.0006(2)	0.0000(5)	-0.0010(5)
OW(2)	0.0030(3)	0.0029(3)	0.0079(9)	0.0003(2)	0.0003(5)	0.0005(5)
F	0.0024(4)	0.0024(3)	0.0060(11)	-0.0009(3)		

*The anisotropic temperature factors are defined as coefficients in $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}\ell^2 + 2\beta_{12}hk + 2\beta_{13}h\ell + 2\beta_{23}kl)]$.

parameters in Table 3. The structure factors are listed in Table 4.[2]

Description of the structure

Topology and geometry of the structure

Minyulite possesses AlO_5F octahedra which share F vertices forming $[Al_2F(H_2O)_4(O_P)_6]$ octahedral dimers, where O_P corresponds to oxygens donated by the phosphate ligands. Two (PO_4) tetrahedra further link the octahedra of each dimer, resulting in a rigid polyhedral cluster with ideal point symmetry *mm*2 and the formula $[Al_2F(H_2O)_4(PO_4)_2(O_P)_2]$. Each cluster shares two octahedral and two tetrahedral vertices with tetrahedra and octahedra of four adjacent clusters, thereby forming sheets oriented parallel to the {001} plane. K^+ cations occupy pockets in the sheets surrounded by four linked clusters. Significant deviation from the ideal 4*mm* symmetry of the sheets is probably necessary to provide a stable coordination for the K^+ cation. Figure 1 depicts the minyulite sheet down the *z* axis. Hydrogen bonds between the water molecules of the octahedra and the unshared vertices of tetrahedra in adjacent sheets represent the only interlayer bonding, accounting for the perfect {001} cleavage observed in minyulite.

Me^{3+} octahedral corner-sharing dimers have not been previously reported in any phosphate mineral species. However, a similar polyhedral cluster has been reported by Fanfani *et al.* (1973) in the structure of copiapite. In this mineral, $Fe^{3+}O_6$ octahedra are joined via a common OH vertex and are further linked via two SO_4 tetrahedra to form a rigid polyhedral cluster topologically equivalent to that in minyulite. Although the ligand environment of Me^{3+} in the two structures is identical, the linkage between clusters is not related. In copiapite, the clusters are linked into chains by sharing vertices of non-cluster (SO_4) tetrahedra (see Fig. 3 of Fanfani *et al.*).

Interatomic distances

Interatomic distances and hydrogen bond angles are given in Table 5. No abnormal average distances are noted, and no distinction between Al–F and Al–O distances is obvious. The electrostatic bond strength sums about the anions are tabulated in Table 6. The bimodal distribution of Al–O bond distances is consistent with the extended electrostatic valence rule of Baur (1970). Al forms long bonds to two oxygens which are each oversaturated with respect to their bond strengths by 0.29 e.s.u. and short bonds to three oxygens which are undersaturated by 0.13, 0.13, and 0.25 e.s.u.

The distance H(1)–H(3) is 1.91(5)A, significantly shorter than the minimum allowable distance of 2.0A suggested by Baur (1972) between hydrogens of different donor groups. This leaves some doubt concerning the accuracy of the hydrogen atom locations.

Hydrogen bonding

One phosphate oxygen, O(4), in minyulite does not directly bond to any other cation. Instead, it achieves approximate electrostatic neutrality by accepting

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[2] To obtain a copy of Table 4, order Document AM-77-039 from the Business office, Mineralogical Society of America, 1909 K Street, N.W., Washington, D.C. 20006. Please remit $1.00 in advance for the microfiche.

four hydrogen bonds from water molecules ligated to the aluminum. Non-linking phosphate oxygens have also been reported in the minerals metavauxite (Baur and Rama Rao, 1968) and seamanite (Moore and Ghose, 1971). The oxygen receives three hydrogen bonds in metavauxite and four in seamanite. The four hydrogen atoms which hydrogen bond to O(4) in minyulite have been located in Figure 1. It can be seen that the four hydrogens and the P^{5+} cation define a tetragonal pyramid about O(4). Such an arrangement was also noted by Baur and Khan (1970) in the crystal structure of $Na_2(H_2O)_7(PO_3OH)$.

Relation to leucophosphite

Moore (1972) showed leucophosphite to possess a structure based on a discrete Fe^{3+} tetramer, idealized in Figure 1 of that publication. The aluminum analog of leucophosphite, prepared by Haseman *et al.* (1950), bears a striking chemical similarity to minyulite; yet, while both structures are based on discrete

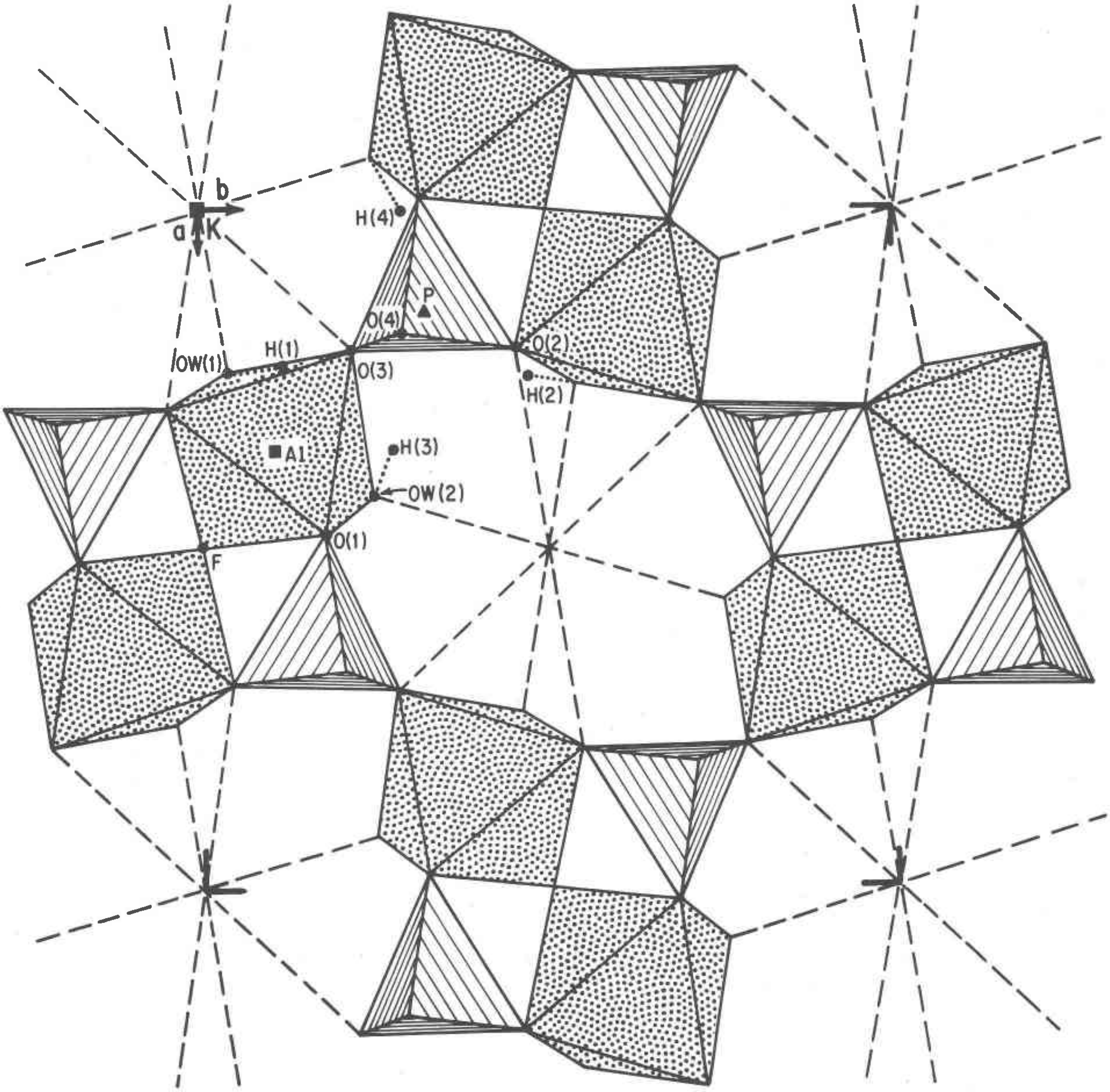

FIG. 1. Polyhedral diagram of the minyulite structure down [001]. The Al–O octahedra are stippled and the P–O tetrahedra are ruled. K–O bonds are shown as dashed lines and OW–H bonds as dotted lines.

TABLE 5. Minyulite. Interatomic distances

Aℓ	
Aℓ - O(3)	1.804(3)Å
- O(1)	1.826(3)
- O(2)	1.844(3)
F	1.851(2)
-OW(2)	1.992(4)
-OW(1)	2.004(4)
average	1.887
O(3)-OW(1)	2.593(5)
F-OW(2)	2.596(3)
F-OW(1)	2.601(3)
F- O(2)	2.631(4)
O(2)- O(3)	2.637(4)
F- O(1)	2.640(4)
O(1)-OW(2)	2.682(5)
O(2)-OW(1)	2.691(5)
O(3)-OW(2)	2.704(4)
OW(1)-OW(2)	2.705(4)
O(1)- O(3)	2.728(4)
O(1)- O(2)	2.744(4)
average	2.663

P	
P - O(3)	1.526(3)
- O(4)	1.531(4)
- O(2)	1.534(3)
- O(1)	1.535(3)
average	1.532
O(1)- O(3)	2.469(4)
O(1)- O(4)	2.481(4)
O(2)- O(4)	2.495(4)
O(2)- O(3)	2.497(4)
O(3)- O(4)	2.528(5)
O(1)- O(2)	2.535(4)
average	2.501

K	
2 K - O(2)	2.855(3)
2 K - O(3)	2.870(3)
2 K -OW(1)	2.874(3)
2 K -OW(2)	2.952(3)
average	2.888

H-bonding	
OW(1)-H(2)	0.76(4)
OW(1)-H(1)	0.81(4)
OW(2)-H(3)	0.96(4)
OW(2)-H(4)	0.98(4)
average	0.88
O(4)-H(3)	1.76(4)
-H(4)	1.88(4)
-H(1)	1.95(4)
-H(2)	2.08(4)
average	1.92
OW(2)-H(3)-O(4)	2.72
OW(1)-H(1)-O(4)	2.76
OW(1)-H(2)-O(4)	2.84
OW(2)-H(4)-O(4)	2.86
average	2.80
OW(2)-O(4)	2.697(4)
OW(1)-O(4)	2.737(4)
OW(2)-O(4)	2.804(4)
OW(1)-O(4)	2.804(4)
average	2.761

Me^{3+} clusters, the clusters are topologically unrelated. Haseman *et al.* synthesized each of these phases from low-temperature aqueous solution. The details of their syntheses appear in Table 7. The formation of these phases can probably be traced to the formation of the particular Me^{3+} cluster in solution. In light of the indication by the structure analysis that the bridging ligand in the minyulite dimer is predominantly F^-, it is particularly noteworthy that F^- was necessary only in the preparation of minyulite. The implication is that the bridging ligand controls the nature of the cluster and, thereby, the phase which will crystallize.

Fe^{3+} and Al^{3+} form octahedral complexes in aqueous solution which commonly condense by sharing $(OH)^-$ ligands. Under the solution conditions noted for the preparation of leucophosphite, the stable cluster in solution is apparently the tetramer. The stabilization of the minyulite dimer in solutions containing F^- is probably the result of a somewhat greater affinity of Al^{3+} for F^- than for $(OH)^-$ and the inability of the tetramer to accommodate F^- as its bridging ligands without severely violating electrostatic valence balance.

TABLE 6. Minyulite. Electrostatic valence balances of cations about anions

	K^+	$Aℓ^{3+}$	P^{5+}	H^+	Σp_O
O(1)		3/6	5/4		1.75
O(2)	1/8	3/6	5/4		1.87
O(3)	1/8	3/6	5/4		1.87
O(4)			5/4	4 x 1/6	1.92
OW(1)	1/8	3/6		2 x 5/6	2.29
OW(2)	1/8	3/6		2 x 5/6	2.29
F		2 x 3/6			1.00

The strong affinity of Al^{3+} for F^- is well known. The octahedral fluoride complexes of aluminum are remarkably stable in aqueous solution. All six species from $[Al(H_2O)_5F]^{2+}$ to $[AlF_6]^{3-}$ are formed. In mineralogical systems, fluoride is often selectively incorporated in phases containing octahedrally-coordinated Al^{3+}. The fluoroaluminates, which crystallize from fluoride-rich aqueous solutions, possess structures based on isolated $[AlF_6]^{3-}$ complexes or on linkages of these complexes. Numerous minerals formed in less fluoride-rich environments incorporate F^- at sites coordinated to two Al^{3+} cations.

The requirement of electrostatic valence balance limits the number of cations which can cluster about an anion. An octahedrally-coordinated Me^{3+} cation contributes bond strengths of 3/6 e.s.u. (electrostatic

TABLE 7. Preparation of minyulite (product T), Al leucophosphite (product HH) and Fe leucophosphite (product I)*

	Minyulite	Aℓ-leucophosphite	Fe-leucophosphite
Phosphate digested	Aℓ	Aℓ	Fe
Cation in solution	K	K	K
$[PO_4^{3-}]$(M)	1.0M	2.0M	1.0-3.5M
$[F^-]$(M)	0.1M	-	-
Final pH	2.8	5.0	2.5-6.0
Temperature (°C)	95	145	75-145
Time (days)	21	50	14-21
Empirical formula	$K_2O \cdot 2A\ell_2O_3 \cdot 2P_2O_5 \cdot 1.6HF \cdot 62H_2O$	$K_2O \cdot 2A\ell_2O_3 \cdot 2P_2O_5 \cdot 5H_2O$	$K_2O \cdot 2Fe_2O_3 \cdot 2P_2O_5 \cdot 5H_2O$

*Haseman *et al.* (1950).

units) to each of its ligands. For a bond configuration to be stable, the charge of the anion must be approximately balanced by the sum of the bond strengths which it receives. It is apparent that perfect electrostatic valence balance is attained when F^- ligates to two Me^{3+} cations, whereas ligation to three Me^{3+} cations would result in severe bond strength oversaturation of F^- by 0.50 e.s.u. A somewhat different situation arises for the $(OH)^-$ anion, since this anion is polar and the hydrogen often forms a weak hydrogen bond with another anion. For electrostatic valence-balance calculations, the O^{2-} and H^+ of the $(OH)^-$ anion are considered separately. Baur (1970) proposed that the O^{2-} receives a bond strength of 5/6 (0.83) e.s.u. from the H^+. On this basis, the ligation of $(OH)^-$ to two Me^{3+} cations results in undersaturation of the anion by 0.17 e.s.u., whereas ligation to three Me^{3+} cations results in oversaturation by 0.33 e.s.u.

Although the minyulite dimer ($2Me^{3+}$/anion) is clearly preferred over the leucophosphite tetramer ($3Me^{3+}$/anion) on the basis of electrostatic valence balance alone when either F^- or $(OH)^-$ acts as the bridging ligand, the preference is considerably greater when the bridging ligand is F^-. The tetramer is apparently stabilized by other factors, which are only outweighed by electrostatic valence imbalance when the bridging ligand is F^-. In Fe^{3+} leucophosphite, the tetramer is probably stabilized by Fe^{3+}–Fe^{3+} spin coupling made possible by the close approach of these cations. This stabilization along with the stronger affinity of Fe^{3+} for $(OH)^-$ than for F^- suggests that the Fe^{3+} analog of minyulite is not likely to exist.

The Al–F–Al linkage is probably also important in the stabilities of a number of other minerals. Fluellite is one of particular interest, since it has been noted in a phosphate-rock paragenesis similar to that of minyulite. The structure of fluellite was shown by Guy and Jeffrey (1966) to be based on F^- corner-sharing chains of Al^{3+} octahedra. Although some substitution of $(OH)^-$ for F^- is indicated by chemical analyses, the bridging ligand is always predominantly F^-. The chain in fluellite represents a greater degree of Al^{3+} octahedral condensation than the dimer in minyulite, and it is likely to result from a greater F^- concentration.

The occurrence of minyulite in soils is strongly dependent on the presence of F^-. Since F^- is generally not prevalent in soils, minyulite is not likely to be important in the fixation of phosphate in soils. Where F^- is locally abundant, however, minyulite (and fluellite) are likely to form in preference to low-temperature aluminum phosphates which do not contain appreciable F^-.

Minyulite has not, as yet, been reported from a pegmatite, although both leucophosphite and fluellite are well-documented as low-temperature species in phosphate-rich pegmatites. This may reflect the improbability of K^+, Al^{3+}, PO_4^{3-}, and F^- all existing in a low-temperature aqueous pegmatitic fluid in sufficient concentrations to permit its formation.

Acknowledgments

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