

Coexisting hydroxyl groups and H₂O molecules in minerals: A single-crystal neutron diffraction study of eosphorite, MnAlPO₄(OH)₂·H₂O

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ABSTRACT

The crystal chemistry of eosphorite from Chamachhu (Skardu District, Baltistan, Pakistan) [(Mn_{0.94}Fe_{0.06}Al_{0.01})_{Σ1.01}AlPO₄(OH)_{1.90}F_{0.10}]_{Σ2}·H₂O, $a = 6.9263(4)$, $b = 10.4356(8)$, $c = 13.5234(10)$ Å, $V = 977.5(1)$ Å³, space group *Cmca*, $Z = 8$], has been reinvestigated by means of electron microprobe analysis in wavelength-dispersive mode and single-crystal neutron diffraction at 20 K. The anisotropic structural refinement has been performed with final agreement index $R_1 = 0.0381$ for 82 refined parameters and 860 unique reflections with $F_o > 4\sigma(F_o)$. The analysis of the difference-Fourier maps of the nuclear density allowed an unambiguous location of the H sites, the description of the H₂O molecule and the OH-group configuration, along with the hydrogen-bonding scheme. We can now describe the structure of eosphorite as built by (Mn,Fe)O₄(OH,H₂O)₂ and AlO₂(OH)₂(OH,H₂O)₂ octahedra, which both form chains running along [100]. The two types of chains are connected, via shared corners, to form a set of (100) sheets held together by P-tetrahedra (and hydrogen bonds) to form a three-dimensional framework. This material provides the rare opportunity to investigate the H-bond configuration of coexisting hydroxyl groups and H₂O molecules in minerals by single-crystal neutron diffraction.

Keywords: Eosphorite, crystal chemistry, single-crystal neutron diffraction, hydrogen bonding

INTRODUCTION

Minerals belonging to the eosphorite [MnAl(PO₄)(OH)₂·H₂O]-childrenite [FeAl(PO₄)(OH)₂·H₂O] series, with the ideal chemical formula M²⁺Al(PO₄)(OH)₂·H₂O, where M²⁺ = Fe²⁺, Mn²⁺ (Hurlbut 1950; Franolet 1980), are widespread secondary phosphates occurring in medium to strongly evolved rare-element granitic pegmatites ranging from the beryl- to the petalite-subtype in the classification of Černý and Ercit (2005). Eosphorite, in particular, is a low-temperature metasomatic mineral in lithium-cesium-tantalum (LCT) granitic pegmatites, where it usually crystallizes in open cavities of pegmatitic dikes (Simmons et al. 2003). Eosphorite is one of the low-*T* alteration products of primary phosphates, mainly lithiophilite. It forms prismatic elongated crystals (up to 10 cm), frequently grouped in divergent clusters of pale-brown or pale-pinkish color.

The unit-cell constants and the possible space groups of childrenite (*Bbam* or *Bba2*) were first reported by Barnes (1949). The crystal structure of childrenite was later solved by Giuseppetti and Tadini (1984) by means of X-ray single-crystal diffraction. The authors refined the crystal structure in the acentric space group *Bba2*, with $a = 10.395(1)$, $b = 13.394(1)$, and $c = 6.918(1)$ Å ($Z = 8$) [childrenite composition: (Mn_{0.11}Fe_{0.89})Al(PO₄)(OH)₂·H₂O].

The crystal structure of eosphorite was first solved by Hanson (1960) in the centrosymmetric *Bbam* space group, with $a \approx 10.52$, $b \approx 13.60$, and $c \approx 6.97$ Å ($Z = 8$), and later

reinvestigated by Hoyos et al. (1993) by means of X-ray single-crystal diffraction, in the *Cmca* space group, with $a = 6.928(1)$, $b = 10.445(1)$, and $c = 13.501(2)$ Å [eosphorite composition: (Mn_{0.76}Fe_{0.24})Al(PO₄)(OH)₂·H₂O]. Hoyos et al. (1993) studied the optical absorption and photoluminescence of the eosphorite sample used for the X-ray investigation.

The crystal structure of both eosphorite and childrenite consists of chains parallel to the *a*-axis (in the *Cmca* space group) that in turn consist of distorted (Mn,Fe)-octahedra, sharing opposite O-O edges, and chains of Al-octahedra. The two types of chains are connected via shared corners to form a set of (100) sheets held together by [PO₄] tetrahedra (and hydrogen bonds) to form a three-dimensional network, as shown in Figure 1.

Although the general structural model of Hoyos et al. (1993) for eosphorite is correct, as shown by bond distances and angles, the quality of the X-ray diffraction data at that time did not allow the authors to provide: (1) a non-ambiguous picture about the Fe/Mn-ordering in the octahedral sites (assuming a fully disordered configuration with one independent Fe/Mn-octahedral site) and (2) the localization of all the proton sites, leaving open questions concerning the topological configuration of the H₂O molecule and of the OH[−] groups, their displacement regime along with the role played by the H-bonds. The location of the proton sites and the nature of the H-bond in this material are important, as the total amount of H₂O is ~16 wt%. In addition, this material provides the rare opportunity to investigate by single-crystal neutron diffraction

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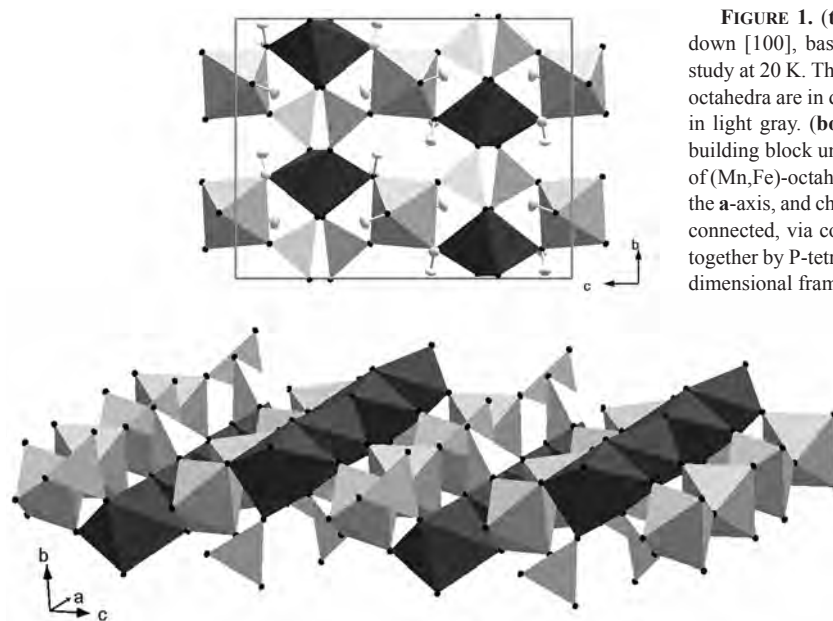


FIGURE 1. (top) The crystal structure of eosphorite viewed down $[100]$, based on the neutron structure refinement of this study at 20 K. Thermal ellipsoid probability factor: 50%. (Mn,Fe) octahedra are in dark gray, Al octahedra in light gray, P tetrahedra in light gray. **(bottom)** Clinographic view of the structure. The building block units of the eosphorite structure consists of chains of (Mn,Fe)-octahedra (sharing opposite O-O edges) running along the a -axis, and chains of Al-octahedra. The two types of chains are connected, via corner-sharing, to form a set of (100) sheets held together by P-tetrahedra (and hydrogen bonds) up to form a three-dimensional framework.

the different role played by H-bonds of coexisting hydroxyl groups and H_2O molecules. In this light, the aim of the present study is a re-investigation of the crystal structure of a natural eosphorite (with low Fe content) by means of low-temperature single-crystal neutron diffraction, to define: (1) the location of the proton sites, and the real topological configuration of the H_2O molecules and OH^- groups, for a full description of the interatomic relationship via H-bond; and (2) the anisotropic displacement parameters of all the atomic sites, H included. The low- T data are useful to reduce thermal libration motion, which could be significant in this class of materials. Since the previous X-ray diffraction studies have proved to be insufficient to directly locate the H atoms, single-crystal neutron diffraction represents, therefore, the best experimental technique that allows to answer the open questions about the crystal structure of this hydrous phosphate.

Samples description and mineralogy

A single crystal of eosphorite up to 14 mm in length and 5 mm in diameter, coming from a granitic pegmatite outcropping in the area of Chamachhu (Changmachhu), Skardu district, Baltistan, Pakistan, was used for this study. The sample comes from a pegmatitic dike located near the little village of Chamachhu, 10 km east of Shengus, along the Indus River on the Gilgit-Skardu road (Blauwet et al. 1997; Kazmi et al. 1985). Eosphorite from Chamachhu occurs as crystals perched on white platy albite (cleavelandite) or on large pollucite crystals (up to 20–30 cm) contained in large miarolitic cavities. The mineralogical association of these pegmatites is fairly evolved and represented by muscovite, lepidolite, spessartine, elbaite, topaz, beryl (gem aquamarine and morganite), pollucite, tantalite-(Mn), fluorapatite, eosphorite, and värynenite. The pegmatitic field is hosted by metamorphic rocks belonging to the Nanga Parbat-Haramosh Massif (Searle 1991).

EXPERIMENTAL METHODS

A prismatic, 14 mm long single-crystal of eosphorite was cut in several fragments used for the chemical analysis and for the neutron diffraction experiment.

Quantitative electron microprobe analysis in wavelength-dispersive mode (EPMA-WDS) was performed on a polished fragment ($1.1 \times 0.8 \times 0.3 \text{ mm}^3$) of the original crystal of eosphorite using a Jeol JXA-8200 electron microprobe at the Earth Science Department of the University of Milano, Italy. The system was operated with an accelerating voltage of 15 kV, a beam current of 5 nA, a counting time of 30 s on the peaks and 10 s on the backgrounds, and a beam diameter of 5 μm . Minerals (graptolite for P, Fe, Mn, and Ca; grossular for Si and Al; K-feldspar for K; forsterite for Mg; omphacite for Na; cancrinite for Cl; and hornblende for F) were used as standards. The raw data were corrected for matrix effects using the $\Phi\rho Z$ method as implemented in the JEOL suite of programs. The average chemical composition and the proportional formula of the investigated eosphorite are given in Table 1.

A second fragment ($3.2 \times 3.5 \times 4.5 \text{ mm}$, Table 2) of the original crystal of eosphorite, free of defect or twinning under the polarized microscope, was used for the neutron diffraction experiment. A preliminary characterization of the single crystal was performed using the Laue backscattering technique as available on the instrument Orient-Express at the Institut Laue-Langevin (ILL), Grenoble (Oulad-diaf et al. 2006). The sample presented broad reflections spots. Consequently, broad scans in omega (up to 6°) were performed for the data collection to get the whole integrated intensities. Intensity data were collected at 20 K on the high-

TABLE 1. Averaged (22 points) WDS electron-microprobe analysis of eosphorite from Chamachhu

	wt%	e.s.d.		apfu
P_2O_5	30.98	0.21	P	1.000
Al_2O_3	22.56	0.07	Al	1.009
FeO	1.92	0.20	Fe^{2+}	0.061
MnO	29.12	0.10	Mn^{2+}	0.940
MgO	0.04	0.02	Mg	0.002
CaO	0.09	0.07	Ca	0.004
Na_2O	0.08	0.01	Na	0.006
* H_2O	14.90		OH^-	1.897
*F	0.86	0.12	F^-	0.103
sum	100.56			
O=F	-0.36		H_2O	1.000
Total	100.20			

Notes: Si, As, K, and Cl were below detection limit; formula proportions calculated on the basis of 1 atom of P per formula unit (apfu); * calculated considering $(\text{OH}+\text{F}) = 2 \text{ apfu}$ and $\text{H}_2\text{O} = 1 \text{ molecule pfu}$.

resolution four-circle diffractometer D9 at ILL using a wavelength of 0.8390(1) Å obtained by reflection from a Cu(220) monochromator. The wavelength was calibrated using a germanium single crystal. D9 is equipped with a small two-dimensional area detector (Lehmann et al. 1989), which for this measurement allowed optimal delineation of the peak from the background. A total number of 2451 reflections (of the type $\pm h, \pm k, \pm l$) were collected up to $2\theta_{\max} = 91.1^\circ$, giving rise to 873 unique reflections (Table 2). For all data, background corrections following Wilkinson et al. (1988) and Lorentz corrections were applied. After corrections, the discrepancy factors among the symmetry related reflections was $R_{\text{int}} = 0.0330$ (Laue class *mmm*) (Table 2).

Least-squares matching of the observed and calculated centroids of the 1078 strongest reflections gave a metrically orthorhombic unit cell with: $a = 6.9263(4)$, $b = 10.4356(8)$, and $c = 13.5234(10)$ Å, with a C-centered lattice. Other details pertaining to the data collections are listed in Table 2. CIF¹ is deposited.

A careful inspection of the diffraction intensities did not show any significant evidence of magnetic ordering in eosphorite structure at 20 K.

¹ Deposit item AM-13-706, CIF. Deposit items are available two ways: For a paper copy contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. For an electronic copy visit the MSA web site at <http://www.minsocam.org>, go to the *American Mineralogist* Contents, find the table of contents for the specific volume/issue wanted, and then click on the deposit link there.

TABLE 2. Details of neutron data collection and refinement of eosphorite

Crystal shape	Prismatic
Crystal size (mm)	$3.2 \times 3.5 \times 4.5$
Crystal color	Translucent pink
Unit-cell constants	$a = 6.9263(4)$ Å $b = 10.4356(8)$ Å $c = 13.5234(10)$ Å $V = 977.5(1)$ Å ³
Chemical formula	MnAl(PO ₄)(OH) ₂ ·H ₂ O
Space group	<i>Cmca</i>
Z	8
T (K)	20
Radiation (Å)	0.8390(1)
Diffractometer	D9 four circle
Data-collection method	ω - θ scans
Max. 2θ (°)	91.1
	$-11 \leq h \leq 8$
	$-11 \leq k \leq 14$
	$-7 \leq l \leq 20$
No. measured reflections	2451
No. unique reflections	873
No. unique refl. with $F_o > 4\sigma(F_o)$	860
No. refined parameters	82
R_{int}	0.0330
R_1 (F) with $F_o > 4\sigma(F_o)$	0.0381
R_1 (F) for all the unique refl.	0.0394
wR_2 (F ²)	0.0689
S	3.240
Weighting scheme: a, b	0.01, 0
Residuals (fm/Å ³)	+0.6/-0.6

Note: $R_{\text{int}} = \sum |F_{\text{obs}} - F_{\text{calc}}| / \sum |F_{\text{obs}}|$; $R_1 = \sum (|F_{\text{obs}}| - |F_{\text{calc}}|) / \sum |F_{\text{obs}}|$; $wR_2 = \{ \sum [w(F_{\text{obs}}^2 - F_{\text{calc}}^2)^2] / \sum [w(F_{\text{obs}}^2)^2] \}^{0.5}$, $w = 1 / [\sigma^2(F_{\text{obs}}) + (a^*P)^2 + (b^*P)^2]$, $P = [\text{Max}(F_{\text{obs}}^2, 0) + 2 * F_{\text{calc}}^2] / 3$.

Structure refinements

The neutron diffraction data of eosphorite were first processed with the program E-STATISTICS, implemented in the WinGX package (Farrugia 1999). The Wilson plot was carried out and the normalized structure factors (E 's) and their statistics of distributions were calculated. The structure of eosphorite was found to be centrosymmetric at 92.9% likelihood. A similar result was obtained by the Sheldrick's $|E^2 - 1|$ criterion (Sheldrick 1997), with $|E^2 - 1| = 0.962$. On the basis of the reflections conditions and of the presence of the inversion center, the crystal structure refinement was then performed in the space group *Cmca* using the SHELX-97 software (Sheldrick 1997), with anisotropic thermal displacement parameters. The starting model was that of Hoyos et al. (1993), without any H site. The neutron scattering lengths of Mn, Fe, Al, P, O, F, and H have been used according to Sears (1986). The secondary isotropic extinction effect was corrected according to Larson's formalism (1967), as implemented in the SHELXL-97 package (Sheldrick 1997). The octahedral M site was modeled with the scattering lengths of Mn and Fe, and the occupancy of the two elements was refined (Table 3). When convergence was achieved, three intense negative residual peaks were found in the final difference-Fourier map of the nuclear density. Further refinement cycles were then performed assigning H to these residual peaks (i.e., H1, H2, and H3 sites; Fig. 2, Table 3), as hydrogen has a negative neutron scattering length (Sears 1986). The final least-square cycles were conducted with anisotropic thermal parameters for all sites including the H-sites. A mixed scattering length of O and F was used to model the O3 and O4 sites. However, this did not improve the figures of merit of the refinement, leading to a not meaningful site occupancy factor of fluorine. When convergence was achieved, all the principal mean-square atomic displacement

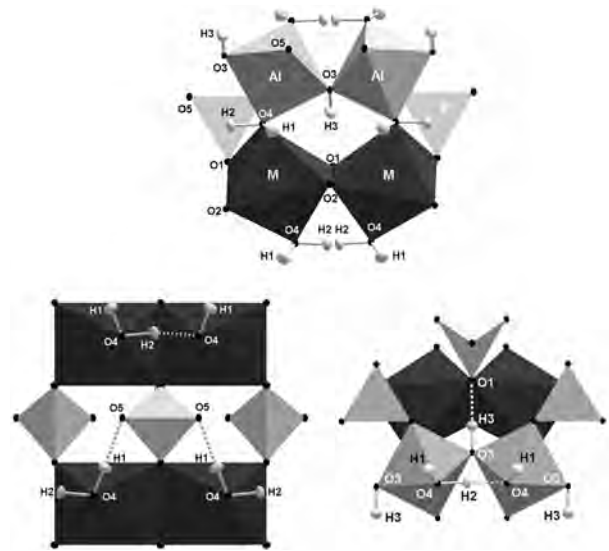


FIGURE 2. Hydrogen location, configuration of OH⁻ groups and H₂O molecules, along with H-bond scheme, in the structure of eosphorite. The occupancy factor of the H2 site is 50%.

TABLE 3. Refined positional and thermal displacement parameters (Å²) and site occupancy factors of eosphorite

Site	s.o.f.	x/a	y/b	z/c	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃	U _{eq}
M	Mn, 0.944(1) Fe, 0.056(1)	0.25	0.13423(14)	0.25	0.0032(3)	0.0055(6)	0.0103(6)	0	-0.0015(3)	0	0.0063(2)
Al	Al, 1	0.25	0.25	0	0.0033(2)	0.0091(5)	0.0066(5)	-0.0012(3)	0.0009(2)	-0.0029(4)	0.0064(2)
P	P, 1	0	0.37739(8)	0.33377(5)	0.0033(2)	0.0061(3)	0.0056(3)	0	0	0.0008(3)	0.0050(1)
O1	O, 1	0	0.26580(7)	0.25871(5)	0.0071(2)	0.0063(3)	0.0058(3)	0	0	-0.0007(2)	0.0064(1)
O2	O, 1	0	0.00716(7)	0.21941(5)	0.0051(2)	0.0059(3)	0.0062(3)	0	0	-0.0011(2)	0.0057(1)
O3	O, 1	0	0.25178(7)	0.04931(5)	0.0037(2)	0.0076(3)	0.0065(3)	0	0	0.0004(2)	0.0059(1)
O4	O, 1	0.18076(5)	0.11057(5)	0.40962(3)	0.0052(1)	0.0069(2)	0.0069(2)	0.0004(1)	-0.0001(1)	-0.0002(2)	0.0063(1)
O5	O, 1	0.31912(5)	0.13209(5)	0.60046(3)	0.0043(1)	0.0076(2)	0.0065(2)	0.0003(1)	-0.0005(1)	0.0013(2)	0.0061(1)
H1	H, 0.990(3)	0.23517(12)	0.02445(11)	0.42046(8)	0.0213(3)	0.0139(5)	0.0225(5)	0.0046(3)	-0.0007(3)	0.0023(4)	0.0192(2)
H2	H, 0.507(3)	0.02866(17)	0.10127(19)	0.41045(13)	0.009(1)	0.020(1)	0.021(1)	-0.002(0)	0	-0.001(1)	0.0165(5)
H3	H, 1	0	0.28409(18)	0.11668(11)	0.0181(4)	0.0349(9)	0.0176(6)	0	0	-0.0073(6)	0.0235(4)

Notes: The anisotropic displacement factor exponent takes the form: $-2\pi^2[(ha^*)^2U_{11} + \dots + 2hka^*b^*U_{12}]$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

TABLE 4. Principal root-mean-square components (R1, R2, and R3, $\times 10^2$ Å) of the atomic displacements parameters

Site	R1	R2	R3	R1/R3
M	10.2(2)	7.4(4)	5.4(3)	1.90
Al	10.6(2)	6.9(4)	5.5(2)	1.94
P	8.1(2)	7.1(2)	5.7(2)	1.41
O1	8.4(1)	8.2(2)	7.3(2)	1.16
O2	8.4(2)	7.1(1)	7.1(2)	1.19
O3	8.8(2)	7.9(2)	6.1(1)	1.44
O4	8.5(1)	8.2(1)	7.1(1)	1.19
O5	9.2(1)	7.6(1)	6.4(1)	1.44
H1	15.4(1)	15.1(2)	10.6(2)	1.45
H2	14.5(3)	14.0(3)	9.4(6)	1.55
H3	19.4(2)	13.4(1)	12.2(3)	1.59

TABLE 5. Relevant bond distances (Å) and angles (°) in the eosphorite structure

M-O1 ($\times 2$)	2.213(1)	O4-H1	0.985(1)
M-O2 ($\times 2$)	2.220(1)	O4-H1*	1.003
M-O4 ($\times 2$)	2.2249(5)	H1...O5	1.757(1)
O1-O1	3.471(1)	O4...O5	2.711(1)
O1-O2	2.751(2)	O4-H1...O5	161.9(1)
O1-O4	2.8908(8)	O4-H2	1.058(1)
O1-O4'	3.563(1)	O4-H2*	1.071
O2-O2	3.561(1)	O4...O4	2.504(1)
O2-O4	3.0163(6)	H2...O4	1.454(1)
O2-O4'	3.0576(8)	O4-H1...O4	170.9(1)
Al-O3 ($\times 2$)	1.8556(3)	H1-O4-H2	107.2(1)
Al-O4 ($\times 2$)	1.9599(5)	O3-H3	0.972(2)
Al-O5 ($\times 2$)	1.8945(5)	O3-H3*	0.995
O3-O4	2.683(1)	O3...O1	2.836(1)
O3-O4'	2.715(1)	H3...O1	1.930(1)
O3-O5	2.614(1)	O3-H3...O1	154.0(1)
O3-O5'	2.689(1)		
O4-O5	2.689(1)	H2...H2	0.397(2)
O4-O5'	2.762(1)		
P-O1	1.545(1)		
P-O2	1.533(1)		
P-O5 ($\times 2$)	1.5396(6)		
O1-O2	2.536(2)		
O1-O5	2.5164(8)		
O2-O5	2.5037(7)		
O5-O5	2.5057(8)		

* Bond distance corrected for "riding motion" following Busing and Levy (1964).

parameters were positively defined and the variance-covariance matrix showed no significant correlation among the refined parameters (i.e., no correlation higher than 0.5). At the end of the last cycle of refinement, no peak larger than $+0.6/-0.6$ fm/Å³ was present in the final difference-Fourier map of the nuclear density (Table 2). The final agreement index (R_i) was 0.0381 for 82 refined parameters and 860 unique reflections with $F_o > 4\sigma(F_o)$ (Table 2). Atomic positions and displacement parameters (U_{ij}) are reported in Table 3. Principal root-mean-square components of the atomic displacements parameters are given in Table 4. Bond lengths and angles are listed in Table 5.

A further test refinement of the structure of eosphorite was performed in the non-centrosymmetric space group $C2cb$. In this case, four independent H sites were located. The refinement converged to the final agreement index $R_i = 0.0314$ (number of observed reflections/number of refined parameters = 9.7), with no residuals larger than $+0.6/-0.5$ fm/Å³. However, when convergence was achieved, the principal mean-square atomic displacement parameters of the (Mn,Fe) site were not positively defined and several other displacement ellipsoids showed a drastically high, and unacceptable, anisotropy. In addition, the variance-covariance matrix showed significant correlations (> 0.82) among the refined parameters.

DISCUSSION AND CONCLUSIONS

The single-crystal neutron structure refinement of this study provides a general structure model in agreement with the previous one reported by Hoyos et al. (1993). The statistics of distributions of the normalized structure factors (E values) and the Wilson plot suggested that the structure is centrosymmetric. This result is corroborated by the structure refinements performed

in the space groups $Cmca$ and $C2cb$. Our experimental findings confirm the previous results reported by Hoyos et al. (1993), who performed a piezoelectricity test on a crystal of eosphorite (from Taquaral, Brazil) showing no evidence of activity in the frequency range from 100 to 1000 KHz, so suggesting the presence of a center of symmetry.

The building block units of the eosphorite structure consist of chains of (Mn,Fe)-octahedra (sharing opposite O-O edges) running along the a -axis, and chains of Al-octahedra. The two types of chains are connected, via corner-sharing, to form a set of (100) sheets held together by P-tetrahedra (and hydrogen bonds) to form a three-dimensional framework, as shown in Figure 1. Distorted channels, confined by a 6-membered ring of polyhedra, run along [100]. The (Mn,Fe)-octahedron is significantly distorted, with $\Delta(O-O)_{\max} \approx 0.81$ Å (i.e., the difference between the longest and the shortest O-O distances) (Table 5), the O-O distance of the shared-edge being significantly shorter than the non-shared ones. We cannot exclude that this is the effect due to the cation-cation repulsion, as M-M ≈ 3.46 Å. The Al-octahedron and the P-tetrahedron appear to be less distorted, with $\Delta(O-O)_{\max} \approx 0.15$ Å and $\Delta(O-O)_{\max} \approx 0.03$ Å, respectively (Table 5).

The low- T structure refinement does not show any evidence of (Mn,Fe)-octahedral ordering, which should lead to a lowering of the symmetry. In addition, the principal root-mean-square components of the atomic displacements parameters (Table 4) do not show any pronounced displacement about the equilibrium position, if compared with those of the other atomic sites, suggesting the absence of a local site splitting. The Fe-content deduced on the basis of the structure refinement is 0.056(1) apfu (Table 3), in good agreement with the chemical analysis (i.e., Fe 0.061 apfu, Table 1). The EPMA-WDS data shows a slight excess of Al (about 0.01 apfu, Table 1), as often reported in eosphorite (e.g., samples from Black Mountain, Maine, and Buckfield, Maine; Hurlbut 1950).

The neutron structure refinement of this study allowed an unambiguous location of the H-sites along with the description of the H-bonding scheme in eosphorite structure. We can now describe the structure as made by (Mn,Fe)₄(OH,H₂O)₂ and AlO₂(OH)₂(OH,H₂O)₂ octahedra (Figs. 1 and 2). The O3 site is the OH⁻ group (i.e., O3-H3) oxygen, whereas O4 is the oxygen of OH⁻ group (i.e., O4-H1) and H₂O molecules (i.e., H2-O4-H1) (Figs. 1 and 2, Table 5). In particular, H₂O molecules have site occupancy of 50%, so that the two H₂O molecules generated by the mirror plane are mutually exclusive (Fig. 2, Table 3). In other words, the two equivalent O4 sites generated by the mirror plane are, respectively, the oxygen of one OH⁻ group and one H₂O molecule, with a local breaking of the symmetry, as shown in Figure 2. Among the three independent Al-O bond distances of the AlO₂(OH)₂(OH,H₂O)₂ octahedron, the Al-OH bond distance (i.e., Al-O3) is the shortest and the Al-(OH,H₂O) distance (i.e., Al-O4) the longest, respectively (Table 5). In the (Mn,Fe)₄(OH,H₂O)₂ polyhedron, the M-(OH,H₂O)₂ bond distance (i.e., M-O4) is the longest (Table 5).

The location of the H⁻ sites and the configuration of the OH⁻ group and H₂O molecules suggested by Hoyos et al. (1993), on the basis of X-ray diffraction data, were not correct: the O3 site was considered as H₂O oxygen atom, whereas the O4 site as OH⁻ group atom.

The geometry of the H₂O molecule and the OH group, along with the hydrogen bonding scheme in eosphorite, are now well defined. The O4-H1 and O4-H2 distances, corrected for “riding motion” (Busing and Levy 1964), are ~1.003 and ~1.071 Å, respectively (Table 5), and two strong hydrogen bonds energetically favorable occur: O4···O5 = 2.711(1) Å, H1···O5 = 1.757(1) Å, and O4-H1···O5 = 161.9(1)°; O4···O4 = 2.504(1) Å, H2···O4 = 1.454(1) Å, and O4-H1···O4 = 170.9(1)° (Fig. 2, Table 5). In other words, symmetry-related O4 act as donor and as acceptor of the H-bond. The H1-O4-H2 angle approaches the ideal value [i.e., 107.2(1); Fig. 2, Table 5] (Chiari and Ferraris 1982; Steiner 1998; Gatta et al. 2008, 2009, 2012). The OH-group shows: O3-H3 distance, corrected for “riding motion,” of ~0.995 Å, O3···O1 = 2.836(1) Å, H3···O1 = 1.930(1) Å, and O3-H3···O1 = 154.0(1)° (Fig. 2, Table 5). The O1 site (i.e., the acceptor of the H bond of the O3-H3 group) is underbonded, as it is shared by two (Mn,Fe)-octahedra and one P-tetrahedra (Fig. 2, Table 5).

The anisotropic structure refinement shows that the principal root-mean-square components of the atomic displacements parameters of the H3 site (belonging to the OH⁻ group) are larger in magnitude than those of the H1 and H2 sites (belonging to the H₂O molecules, Table 4).

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Gatta et al.: Crystal chemistry of eosphorite, American Mineralogist
Supplemental data, AM-13-706.

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 O1 Mn P 96.24(2) . 14_546 ?
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 O4 Mn P 97.27(4) . 14_546 ?
 O5 Mn P 137.495(11) 13_556 14_546 ?
 O5 Mn P 117.886(15) 16 14_546 ?
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 P Mn P 117.307(7) . 14_546 ?
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 O1 Mn P 150.86(3) . 5_545 ?
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 O4 Mn P 72.41(3) . 5_545 ?
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 O4 Al P 105.53(2) 4 6_454 ?
 O4 Al P 74.47(2) 16 6_454 ?
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 O4 Al Al 104.163(11) 4 7_455 ?
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 O2 P O1 110.95(6) 14_556 . ?
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 O4 O5 O4 91.95(2) 13_556 . ?
 O3 O5 O4 59.713(17) 10_656 . ?
 O4 O5 O4 75.78(2) 3_556 . ?
 P O5 Fe1 78.81(3) 13_556 13_556 ?
 Al O5 Fe1 85.44(2) 4 13_556 ?
 O2 O5 Fe1 96.37(3) 2 13_556 ?
 O5 O5 Fe1 98.593(7) 11_655 13_556 ?
 O1 O5 Fe1 43.481(15) 13_556 13_556 ?
 O3 O5 Fe1 71.81(2) 6_455 13_556 ?
 O4 O5 Fe1 43.27(2) 13_556 13_556 ?
 O3 O5 Fe1 101.04(3) 10_656 13_556 ?
 O4 O5 Fe1 133.62(2) 3_556 13_556 ?
 O4 O5 Fe1 126.85(2) . 13_556 ?
 P O5 Mn 78.81(3) 13_556 13_556 ?
 Al O5 Mn 85.44(2) 4 13_556 ?
 O2 O5 Mn 96.37(3) 2 13_556 ?
 O5 O5 Mn 98.593(7) 11_655 13_556 ?
 O1 O5 Mn 43.481(15) 13_556 13_556 ?
 O3 O5 Mn 71.81(2) 6_455 13_556 ?
 O4 O5 Mn 43.27(2) 13_556 13_556 ?
 O3 O5 Mn 101.04(3) 10_656 13_556 ?
 O4 O5 Mn 133.62(2) 3_556 13_556 ?
 O4 O5 Mn 126.85(2) . 13_556 ?
 Fe1 O5 Mn 0.0 13_556 13_556 ?

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_diffraction_reflections_theta_full	45.55
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_refinement_diff_density_rms	0.150