Fanfaniite, $Ca_4Mn^{2+}Al_4(PO_4)_6(OH,F)_4 \cdot 12H_2O$, a new mineral with a montgomeryite-type structure

Ian E. GREY[1,*], Anthony R. KAMPF[2], Jason B. SMITH[3], Colin M. MacRAE[1] and Erich KECK[4]

[1]CSIRO Mineral Resources, Private Bag 10, Clayton South, Victoria 3169, Australia
*Corresponding author, e-mail: ian.grey@csiro.au
[2]Department of Mineral Sciences, Natural History Museum of Los Angeles County,
900 Exposition Boulevard, Los Angeles, CA 90007, USA
[3]2148 McClintock Rd., Charlotte, NC 28205, USA
[4]Algunderweg 3, Etzenricht 92694, Germany

Abstract: Fanfaniite, $Ca_4Mn^{2+}Al_4(PO_4)_6(OH,F)_4 \cdot 12H_2O$, is a new secondary phosphate mineral from the Foote spodumene mine, North Carolina, USA and the Hagendorf-Süd pegmatite, Bavaria, Germany. At the Foote mine, it forms radial aggregates up to 0.5 mm in diameter of colourless, transparent, thin blades, flattened on {010} and elongated on [001], associated with whiteite-(CaMnMn). At Hagendorf-Süd, the mineral occurs as isolated very thin laths on the surface of fibrous spheroids of kayrobertsonite and is associated with altered triplite–zwieselite and whiteite-(CaMnMn). The measured density (Foote mine) is 2.58(2) g cm^{-3}. Optically, fanfaniite (Foote mine) is biaxial (–), with $\alpha = 1.573(2)$, $\beta = 1.582(2)$, $\gamma = 1.585(2)$ and $2V$(meas) = 57(1)°. Dispersion was not observed. The optical orientation is: $Z = \mathbf{b}$, $X \wedge \mathbf{c} \approx 40°$ in β obtuse. Pleochroism was not evident. Electron microprobe analyses gave the empirical formulas $Ca_{3.91}Mn^{2+}_{0.77}Mg_{0.10}Zn_{0.02}Al_{3.89}Fe^{3+}_{0.21}(PO_4)_6(OH)_{3.90}(H_2O)_{12.10}$ (Foote mine) and $Ca_{3.73}Mn^{2+}_{0.76}Mg_{0.25}$ $Zn_{0.08}Al_{3.89}Fe^{3+}_{0.29}(PO_4)_6F_{1.10}(OH)_{3.08}(H_2O)_{11.82}$ (Hagendorf-Süd). Fanfaniite has monoclinic symmetry, space group $C2/c$, with $a = 10.021(4)$ Å, $b = 24.137(5)$ Å, $c = 6.226(3)$ Å, $\beta = 91.54(2)°$ and $V = 1505(1)$ Å^3. The crystal structure was refined to $R_{obs} = 0.043$ for 1909 unique reflections to a resolution of 0.7 Å. Fanfaniite is the Mn^{2+}-dominant analogue of montgomeryite. The name honours Luca Fanfani who structurally characterised many phosphate minerals including montgomeryite.

Key-words: fanfaniite; new mineral; phosphate; Foote mine, North Carolina; Hagendorf-Süd pegmatite, Bavaria; crystal structure; montgomeryite-type structure.

1. Introduction

The minerals calcioferrite (Blum, 1858), montgomeryite (Larsen, 1940), kingsmountite (Dunn *et al.*, 1979) and zodacite (Dunn *et al.*, 1988) have in common the same general formula, $Ca_4AB_4(PO_4)_6(OH)_4 \cdot 12H_2O$ and similar powder XRD patterns, that can be indexed with C-centred monoclinic unit cells, $a \sim 10.1$, $b \sim 24.1$, $c \sim 6.3$ Å and $\beta \sim 91.2°$. A and B are divalent and trivalent cations, respectively, with A^{2+} and B^{3+} = Mg and Fe for calcioferrite, Mg and Al for montgomeryite, Fe^{2+} and Al for kingsmountite and Mn and Fe for zodacite. Montgomeryite was the first of these minerals to have the structure determined (Moore & Araki, 1974), in space-group $C2/c$, although Fanfani *et al.* (1976) proposed that the correct space group was $C2$. Recently a high-quality refinement of the calcioferrite structure was made in $C2/c$ (Lafuente *et al.*, 2014).

A Mn^{2+} analogue of kingsmountite, $Ca_4Fe^{2+}Al_4(PO_4)_6(OH)_4 \cdot 12H_2O$ (Dunn *et al.*, 1979), was first reported by Mücke (1987) from the Hagendorf-Süd pegmatite mine. Mücke examined spherules of white needle crystals in druses of altered zwieselite collected by E. Keck from 60 to 67 m level of the mine and confirmed that it had the same powder X-ray diffraction (PXRD) pattern as that for kingsmountite. On the basis of energy-dispersive X-ray analysis, he proposed the formula $(Ca,Zn)_4MnAl_4$ $(PO_4)_6(OH,Cl)_4 \cdot 12H_2O$ and referred to the mineral as kingsmountite-Mn. In a later study of minerals provided by E. Keck from 60 to 67 m level of the Hagendorf-Süd mine (Grey *et al.*, 2010), the same mineral was identified and characterized by electron microprobe (EMP) analyses. The crystals, however, were not of sufficient diffracting quality to enable the crystal structure to be refined. Subsequently, one of us (JS) obtained a sample of the Mn^{2+} analogue of kingsmountite from the Foote mine, North Carolina that contained crystals of good diffraction quality. This allowed the crystal structure to be refined and a new mineral proposal to be prepared.

The new mineral and its name, fanfaniite, were approved by the Commission on New Minerals, Nomenclature and Classification (CNMNC) of the International Mineralogical Association (IMA2018-053). The name fanfaniite honours

Italian geochemist/mineralogist/crystallographer Luca Fanfani (born 1941). Some of Professor Fanfani's earliest research, while a Professor of Mineralogy at the University of Perugia, focused on structure determinations and crystallochemical classification of sulfate and phosphate minerals. Between 1970 and 1976, he was among the most prolific and influential mineralogical crystallographers studying these mineral classes. His work on phosphates includes important studies on beraunite, fairfieldite, roscherite, spencerite, strunzite, switzerite, wardite and, most importantly in the context of this proposal, montgomeryite (Fanfani *et al.*, 1976). Since 1976, he has been Professor of Mineralogy at the University of Cagliari, conducting research on processes involving the interaction of minerals and water, with specific focus on understanding the transport of toxic elements in near-surface environments. In 2012, Professor Fanfani was recognized with The Distinguished Service Award from the International Association of Geochemistry.

The Foote mine and Hagendorf-Süd occurrences of fanfaniite are co-type localities. One holotype specimen and one cotype specimen from the Foote mine are housed in the mineralogical collections of the Natural History Museum of Los Angeles County, catalogue numbers 66771 (holotype) and 66772 (cotype). The Hagendorf-Süd cotype specimen, attached to kayrobertsonite, is housed in the collections of Museum Victoria, registered number M48795. Apart from EMP analyses, the results are all for the Foote-mine holotype specimen.

2. Occurrence and associated minerals

Fanfaniite was found in thin solution fractures and small vugs of partially oxidized pegmatite on the East dump of the Foote Lithium Company mine, Kings Mountain district, Cleveland County, North Carolina, USA (35°12′40″ N, 81°21′20″ W). The Foote Lithium Company mine is the type locality for 14 other minerals (*e.g.* Atencio *et al.*, 2008; Mills *et al.*, 2016). Fanfaniite is associated with whiteite-(CaMnMn) and/or rittmannite at the Foote mine.

Fanfaniite was also found in specimens collected by EK between 60 and 67 m levels of the mine at the Hagendorf-Süd pegmatite, Hagendorf, Oberpfalz, Bavaria, Germany (49°39′1″ N, 12°27′35″ E). Hagendorf-Süd is the type locality for 25 minerals of which 24 are secondary phosphates. At Hagendorf-Süd, fanfaniite occurs as isolated, very thin laths distributed on the surface of kayrobertsonite, as described by Grey *et al.* (2010). In that paper, kayrobertsonite (Mills *et al.*, 2016) was referred to as a fibrous form of nordgauite. Other associated minerals are altered triplite–zwieselite, whiteite-(CaMnMn), fluorapatite, nordgauite, morinite, fluellite and Al-bearing strunzite.

3. Physical and optical properties

Fanfaniite from the Foote mine forms radial aggregates of thin, translucent white blades (Fig. 1) with widths ranging from 20 to 50 μm, lengths up to 200 μm and thickness

Fig. 1. Fanfaniite from the Foote mine. Field of view is 0.50 mm. Natural History Museum of Los Angeles County, catalogue number 66771.

generally <10 μm. The blades are flattened on {010} and elongated on [001], with {010} the only discernible form. The blades have a pearly lustre and are flexible and elastic, with good cleavage on {010}. The measured density is 2.58 (2) g cm^{-3} determined by flotation in a mixture of methylene iodide and toluene. The calculated density is 2.58 g cm^{-3}, based on the empirical formula and single-crystal XRD cell.

Optically, fanfaniite is biaxial (–), with the indices of refraction $\alpha = 1.573(2)$, $\beta = 1.582(2)$, $\gamma = 1.585(2)$, measured in white light. The $2V$(meas) = 57(1)° from extinction data using EXCALIBR (Gunter *et al.*, 2004); $2V$(calc.) = 59.7°. Dispersion was not observed. The optical orientation is: $Z = \mathbf{b}$, $X \wedge \mathbf{c} \approx 40°$ in β obtuse. Pleochroism was not evident. The Gladstone–Dale compatibility, $1 - (K_P/K_C)$, (Mandarino, 2007) is −0.019 (excellent) based upon the empirical formula, density calculated using the single-crystal cell and the measured indices of refraction.

4. Chemical composition

Crystals of fanfaniite were analysed using wavelength-dispersive spectrometry on a JEOL JXA 8500F Hyperprobe operated at an accelerating voltage of 15 kV and a beam current of 4 nA. The beam was defocused to 4 μm. Analytical results are given in Table 1. There was insufficient material for direct determination of H_2O content so it was based upon the crystal structure analysis. The low analysis totals are most likely due to the electron beam penetrating the very thin blades.

The empirical formula for the Foote mine sample, normalised to 6 P and 40 anions, with OH adjusted for charge balance, is $Ca_{3.91}Mn^{2+}_{0.77}Mg_{0.10}Zn_{0.02}Al_{3.89}Fe^{3+}_{0.21}$ $(PO_4)_6(OH)_{3.90}(H_2O)_{12.10}$. Hagendorf-Süd fanfaniite contains minor F substituting for OH, which was not detected in the Foote mine material. The empirical formula for Hagendorf-Süd material is, $Ca_{3.73}Mn^{2+}_{0.76}Mg_{0.25}Zn_{0.08}Al_{3.89}Fe^{3+}_{0.29}$ $(PO_4)_6F_{1.10}(OH)_{3.08}(H_2O)_{11.82}$. A similar formula for

Table 1. Analytical data (wt%) for fanfaniite.

Constituent	Foote mine (average of 14)			Hagendorf-Süd	Probe standard
	Mean	Range	SD	Average of eight (SD in parentheses)	
ZnO	0.15	0–0.39	0.15	0.52 (0.30)	Phosphophyllite
MnO	4.51	3.85–4.78	0.32	4.26 (0.85)	Rhodonite
MgO	0.33	0.20–0.51	0.10	0.85 (0.14)	Spinel
CaO	18.10	17.55–18.72	0.35	16.5 (1.5)	Wollastonite
Al_2O_3	16.39	15.27–17.06	0.45	15.6 (1.0)	Berlinite
Fe_2O_3	1.39	0.85–2.43	0.45	1.84 (0.18)	Hematite
P_2O_5	35.14	34.12–36.44	0.80	33.50 (0.85)	Berlinite
F	n.d.			1.64 (0.29)	
–O=F				−0.69	
Σoxides*	76.01			74.02	

n.d., not detected; SD, standard deviation.*H_2O not analysed = 21.5 wt% from structure.

fanfaniite associated with whiteite-(CaMnMn) at Hagendorf-Süd has been reported by Yakovenchuk *et al.* (2012).

The ideal formula is $Ca_4MnAl_4(PO_4)_6(OH,F)_4 \cdot 12H_2O$, which for the F-free phase requires CaO 19.05, MnO 6.03, Al_2O_3 17.32, P_2O_5 36.17, H_2O 21.41, total 100 wt%.

5. X-ray crystallography

Powder X-ray diffraction data for the Foote mine sample were obtained using a Rigaku R-AXIS Rapid II curved imaging plate microdiffractometer, with monochromatized Mo$K\alpha$ radiation. A Gandolfi-like motion on the φ and ω axes was used to randomize the sample. Observed *d* values and intensities were derived by profile fitting using JADE 2010 software. Data (in Å for Mo$K\alpha$) are given in Table S1 of the Supplementary Material linked to this article and freely available at https://pubs.geoscienceworld.org/eurjmin/. Unit-cell parameters refined from the powder data using JADE 2010 with whole-pattern fitting are $a = 10.039(5)$ Å, $b = 24.198(5)$ Å, $c = 6.261(5)$ Å, $\beta = 91.50(2)°$ and $V = 1520.4(15)$ Å.

A single-crystal data collection was made at the macromolecular beam line MX2 of the Australian Synchrotron. Data were collected using monochromatic radiation with a wavelength of 0.7107 Å. The crystal was maintained at 100 K in an open-flow nitrogen cryostream. Further data collection details are given in Table 2. Indexing of the single-crystal data was achieved with a *C*-centred monoclinic cell, with reflection extinctions consistent with a *c*-glide plane. The normalised structure factor statistics strongly supported centrosymmetry ($|E^2–1| = 0.97$, *cf.* 0.968 for centrosymmetric and 0.736 for non-centrosymmetric), confirming the space group as *C*2/*c*.

6. Crystal-structure refinement

The published non-H atom coordinates for calcioferrite (Lafuente *et al.*, 2014) were used as a starting model, although a single Ow3 site was used in place of the split site reported by Lafuente *et al.* (2014). The structure was refined using JANA2006 (Petříček *et al.*, 2014). Refinement of

metal atom site occupancies indicated some minor substitution of Fe^{3+} into one of the two independent Al sites (Al2). The EMP-determined atoms per formula unit of Mn and Mg, together with Fe were incorporated and fixed in the Mn site. Refinement with anisotropic displacement parameters for all atoms converged at $wR_{obs} = 0.060$ for 1854 reflections with $I > 3\sigma(I)$. The water molecule Ow3 was found to have high anisotropic U_{ij} values and we examined the possibility of splitting Ow3, as reported by Lafuente *et al.* (2014). Removing Ow3 from the refinement and relocation in a difference Fourier map did not provide clear evidence for a split atom. At this stage, five H atoms were located in a difference-Fourier map, in positions similar to those reported by Lafuente *et al.* (2014). The H atom positions were refined subject to the soft constraints of O–H = 1.85(1) Å and H–O–H = 109.47(1)°. A common isotropic displacement parameter was refined for the five H atoms.

We checked the possibility of the space group being *C*2, as reported for montgomeryite by Fanfani *et al.* (1976). The half-occupied Mn site in *C*2/*c* splits into two sites in *C*2 and only one of these is occupied in the case of montgomeryite. A structure solution in *C*2, with merohedral twinning, was obtained using SHELXT (Sheldrick, 2015). It refined to a similar *R* factor as the *C*2/*c* model, but it was found that the two independent Mn sites were each half-occupied as in the *C*2/*c* model. Ordering of Mn into one or other of the sites increased *R* from 0.05 to 0.08.

Details of the data collection and refinement are given in Table 2. The refined coordinates, equivalent isotropic displacement parameters and bond valence sum (BVS) values (Gagné & Hawthorne, 2015) from the single-crystal refinement are reported in Table 3. Anisotropic displacement parameters are given in Table S2 of the Supplement. Polyhedral bond distances are reported in Table 4 and the H-bonding scheme in Table 5. We could not find unambiguous H positions associated with Ow3.

7. Discussion

A [001] projection of the fanfaniite structure is given in Fig. 2. The structure has been described in detail for

Table 2. Crystal data and structure refinement for fanfaniite from the Foote mine.

Formula (ideal)	$Ca_4MnAl_4(PO_4)_6(OH,F)_4 \cdot 12H_2O$
Formula weight	1177.0
Temperature	100 K
Wavelength	0.7107 Å
Space group	$C2/c$
Unit-cell dimensions	$a = 10.021(4)$ Å
	$b = 24.137(5)$ Å
	$c = 6.226(3)$ Å
	$\beta = 91.54(2)°$
Volume	1505(1) Å^3
Z	2
Absorption coefficient	1.71 mm^{-1}
Absorption correction	SADABS, $T_{min}/T_{max} = 0.62/0.75$
Crystal size	$0.025 \times 0.06 \times 0.005$ mm^3
θ range for data collection	2.2–32.0°
Index ranges	$-14 \leq h \leq 14, -32 \leq k \leq 32,$
	$-7 \leq l \leq 7$
Data resolution for refinement	0.7 Å
Reflections collected	13,848
Independent reflections	1909
Reflections with $I_o > 3\sigma(I)$	1869
Refinement method	Full-matrix least-squares on F
Restraints/constraints/parameters	7/19/149
Final R indices $[I > 3\sigma(I)]$	$R_{obs} = 0.042$, $wR_{obs} = 0.057$
R indices (all data)	$R_{obs} = 0.043$, $wR_{obs} = 0.058$
Largest diff. peak and hole	0.84 and -0.80 e Å^{-3}

Table 3. Atom coordinates, equivalent isotropic displacement parameters (Å^2) and bond-valence sums (BVS) for fanfaniite from the Foote mine.

	x	y	z	U_{eq}	BVS
Ca1	0	0.06077(3)	0.25	0.0173(2)	1.82
Ca2	0	0.33045(3)	0.25	0.0149(2)	2.12
Al1	0.25	0.25	0	0.0126(3)	3.08
Al2*	0	0.17112(4)	0.75	0.0133(3)	2.94
Mn**	0	0.47293(5)	0.25	0.0206(3)	2.23
P1	0.5	0.30003(3)	0.75	0.0131(2)	5.02
P2	0.25905(6)	0.11577(2)	0.9603(1)	0.0151(2)	4.95
O1	0.6173(2)	0.26095(7)	0.7073(3)	0.0164(5)	1.98
O2	0.4703(2)	0.33741(7)	0.5557(3)	0.0166(5)	1.84
O3	0.3103(2)	0.17504(7)	0.0111(3)	0.0168(5)	1.91
O4	0.3761(2)	0.08755(7)	0.8517(3)	0.0183(5)	1.79
O5	0.1374(2)	0.11758(7)	0.7998(3)	0.0188(5)	1.71
O6	0.2194(2)	0.08679(8)	0.1660(3)	0.0254(6)	1.63
Oh1	0.3704(2)	0.27164(7)	0.2203(3)	0.0153(5)	1.05
Ow1	0.1645(2)	0.32854(9)	0.5252(4)	0.0253(6)	0.34
Ow2	0.1126(2)	0.02580(7)	0.5817(3)	0.0191(5)	0.43
Ow3	0.1226(2)	0.4737(1)	0.5683(5)	0.052(1)	0.31
H12	0.237(4)	0.319(2)	0.454(8)	0.070(7)	
H22	0.190(3)	0.016(2)	0.544(9)	0.070(7)	
H11	0.187(4)	0.351(2)	0.625(8)	0.070(7)	
H21	0.119(4)	0.053(2)	0.674(8)	0.070(7)	
H1	0.347(5)	0.262(2)	0.340(6)	0.070(7)	

*Occupancy 0.960(6)Al + 0.040Fe.
**Occupancy 0.76Mn + 0.12Mg + 0.12Fe.

montgomeryite by Moore & Araki (1974) and only the main features are summarised here. Fanfaniite has a heteropolyhedral layer structure parallel to (010), based

Table 4. Polyhedral bond lengths (Å) for fanfaniite from the Foote mine.

Ca1–O2 ×2	2.752(2)	Ca2–O1 ×2	2.517(2)
Ca1–O6 ×2	2.359(3)	Ca2–O3 ×2	2.537(2)
Ca1–Ow2 ×2	2.475(3)	Ca2–O4 ×2	2.429(2)
Ca1–Ow2 ×2	2.608(2)	Ca2–Ow1 ×2	2.346(3)
Av.	2.548	Av.	2.457
Al1–O1 ×2	1.897(2)	Al2–O2 ×2	1.945(3)
Al1–O3 ×2	1.908(2)	Al2–O5 ×2	1.908(2)
Al1–Oh1 ×2	1.876(2)	Al2–Oh1 ×2	1.902(2)
Av.	1.894	Av.	1.918
Mn–O4 ×2	2.029(2)	P2–O3	1.550(2)
Mn–Ow3 ×2	2.304(4)	P2–O4	1.530(2)
Mn–Ow3 ×2	2.127(3)	P2–O5	1.556(2)
Av.	2.153	P2–O6	1.521(2)
		Av.	1.539
P1–O1 ×2	1.536(2)		
P1–O2 ×2	1.532(2)		
Av.	1.534		

Table 5. Hydrogen bonds in fanfaniite (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D-A$	$D-H \cdots A$
Ow1–H11···O3	0.86(5)	2.35(5)	2.892(4)	121(4)
Ow1–H11···O6	0.86(5)	2.18(5)	3.018(3)	167(4)
Ow1–H12···O2	0.89(4)	2.45(4)	3.073(4)	127(4)
Ow1–H12···Oh1	0.89(4)	2.31(5)	3.156(3)	160(4)
Ow2–H21···O5	0.88(5)	1.74(5)	2.607(3)	166(5)
Ow2–H22···Ow3	0.84(4)	2.04(4)	2.837(4)	157(5)
Oh1–H1···Ow1	0.82(4)	2.34(5)	2.917(3)	127(5)

on [100] *zig-zag* chains of corner-connected Al-centred octahedra that are linked along [001] *via* corner-sharing with PO_4 tetrahedra. The eight-coordinated Ca2 polyhedra form part of the layer structure, while the eight-coordinated Ca1-centred polyhedra and Mn-centred octahedra occupy sites on the surface of the layers. The $Ca1O_4(H_2O)_4$ polyhedra share edges through the water molecules Ow2 (Fig. 2) to form chains along [001]. The Mn site has only half-occupancy, as also reported for montgomeryite (Moore & Araki, 1974) and calcioferrite (Lafuente *et al.*, 2014). The BVS calculations are consistent with the minor iron content at the Mn site being Fe^{3+}. The H-bonding is identical to that reported in calcioferrite by Lafuente *et al.* (2014).

Fanfaniite is the Mn analogue of montgomeryite, with Mn replacing Mg in the *A* site. Comparative data for the two minerals are given in Table 6. Fanfaniite is also closely related to kingsmountite, which has $A = Fe^{2+}$ (Dunn *et al.*, 1979). However, in a re-investigation of the holotype specimen of kingsmountite, we found that lowering of the symmetry to triclinic results in splitting of the Ca sites into four independent sites, one of which is dominated by Mn.
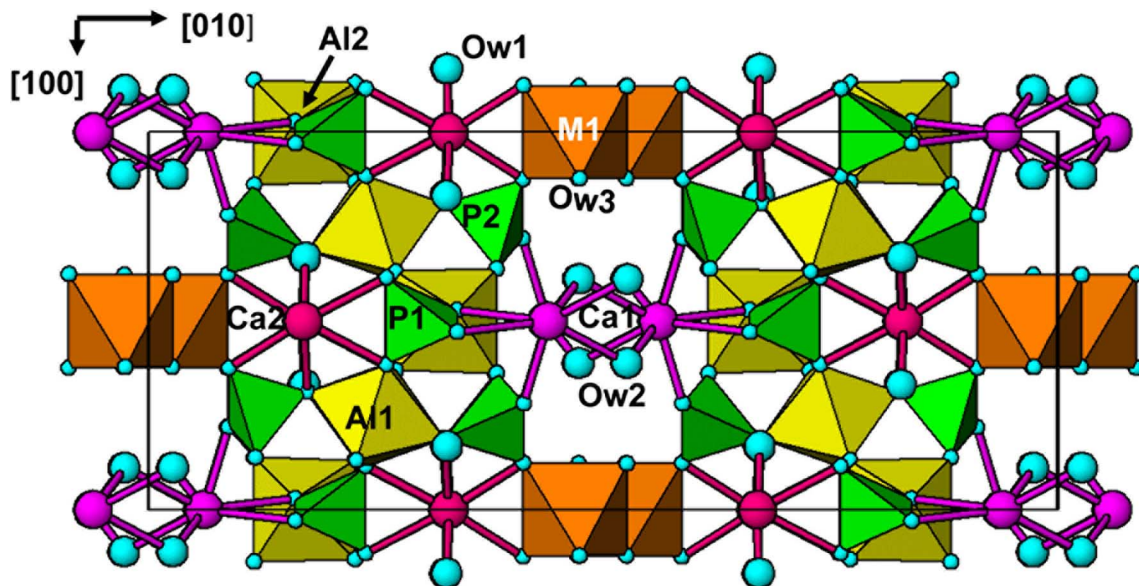
Fig. 2. Fanfaniite structure, projected along [001]. M1 = dominant Mn.

Table 6. Comparative data for fanfaniite and montgomeryite.

	Fanfaniite	Montgomeryite
Ideal formula	$Ca_4Mn^{2+}Al_4(PO_4)_6(OH,F)_4 \cdot 12H_2O$	$Ca_4MgAl_4(PO_4)_6(OH)_4 \cdot 12H_2O$
Crystal system	Monoclinic	Monoclinic
Space group	$C2/c$	$C2/c$ (or $C2$)
a (Å)	10.021(4)	10.023(1)
b (Å)	24.137(5)	24.121(3)
c (Å)	6.226(3)	6.243(1)
β (°)	91.54(2)	91.55(1)
V (Å^3)	1505(1)	1509
Z	2	2
Strongest	12.14 (33) (020)	12.0 (9) (020)
lines in	5.13 (97) (111)	5.10 (10) (200)
X-ray	3.137 (28) (260)	3.125 (5) (002)
powder	2.938 (100) ($\bar{3}$11, $\bar{1}$71)	2.948 (4) (311), 2.893 (9) (171)
pattern	2.618 (70) (202)	2.612 (7) (222)
d (I) (hkl)	2.249 (25) ($\bar{3}$71, 172)	2.244 (5) (not indexed)
	1.740 (22) (313, 462)	
D_{meas} (g cm^{-3})	2.58(2)	2.530(5)
Opt. character	Biaxial (–)	Biaxial (–)
α	1.573(2)	1.572(2)
β	1.582(2)	1.578(2)
γ	1.585(2)	1.582(2)
$2V_{meas}$ (°)	57(1)	75(10)
Reference	This study	PXRD: Moore (1964)
		Unit cell: Moore & Araki (1974)
		Density, optics: Larsen (1940)

Note added in proof: The calcioferrite group, of which fanfaniite is a new member, has been formally approved, CNMNC 19-B. A revised formula for

kingsmountite, $Ca_3MnFeAl_4(PO_4)_6(OH)_4 \cdot 12H_2O$, has also been approved.

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