X-Ray Powder Diffraction Data for Wolfeite: $(Fe_{0.59}Mn_{0.40}Mg_{0.01})_2PO_4(OH)$

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Abstract
Wolfeite $(Fe_{0.59}Mn_{0.40}Mg_{0.01})_2PO_4(OH)$ from the Hagendorf-Sud pegmatite, Bavaria, Federal Republic of Germany, yields unit-cell parameters of: a = 12.319(1), b = 13.230(2), c = 9.840(1) Å and β = 108° 24(1)'. $D_{meas.}$ = 3.82(2); $D_{calc.}$ =3.88. An indexed powder diffraction pattern is given.

Introduction

During a study of the Fe,Mn phosphate minerals occurring in granitic pegmatites, and particularly of those belonging to the triploidite-wolfeite isomorphous series, we noticed the absence of recent X-ray powder diffraction data on wolfeite, $(Fe,Mn)_2PO_4(OH)$.

The specimen analyzed was collected by one of us (A.M.F.) in the Hagendorf-Sud pegmatite, Bavaria, Federal Republic of Germany. Wolfeite is brown to pinkish-brown and is associated with triphylite, hagendorfite, arrojadite, and hematite as previously described by Strunz *et al.* (1975).

The objective of this paper is to provide X-ray powder diffraction data for wolfeite, to calculate the unit-cell parameters and to check its chemical formula by comparison of the measured and calculated density.

X-Ray Diffraction Data

Frondel (1949) published an unindexed powder pattern of wolfeite, which was reproduced and indexed on PDF 5-612. No other powder diffraction data were found.

A diffractogram was recorded with a diffractometer equipped with a graphite diffracted-beam monochrometor, using Fe radiation (λFeKα = 1.9373 Å), and a scanning speed of 1/2° 2θ/min. The d-spacings were corrected with an internal standard of $Pb(NO_3)_2$ (a = 7.8568 Å). Indexing was performed taking into account the intensities of reflections as measured for the crystal structure determination of triploidite (Waldrop, 1970) and the intensities computed with the program Lazy Pulverix (Yvon *et al.*, 1977). Least-squares refinement using the program of Appleman and Evans (1973) yielded unit cell parameters for wolfeite from Hagendorf-Sud: a = 12.319 (1), b = 13.230(2), c = 9.840(1) Å, β = 108° 24(1)', and V=1521.9(2) Å^3 in space group $P2_1/a$(14). X-ray powder diffraction data are given in Table 1. The indexing is in good agreement with that given for triploidite, $(Mn_{1.5}Fe_{0.5})PO_4(OH)$, from Branchville, Connecticut (PDF 26-1239).

Table 1.
X-ray Powder Diffraction Data of Wolfeite. Feλ = 1.9373Å

Z00286 **$Fe_2PO_4(OH)$**
Iron Phosphate Hydroxide Wolfeite

Rad. FeKα λ 1.9373 **Filter** Mono. **d-sp** Diff.
Cut off **int.** Diffractometer **I/Icor.**
Ref Antenucci, D., Fontan, F., Fransolet, A.-M. (to be published)., *Powder Diffraction Journal*

Sys. Monoclinic **S.G.** $P2_1/a$ (14)
a 12.319(1) **b** 13.230(2) **c** 9.840(1) **A** 0.9311 **C** 0.7438
α β 108.40(1) γ **Z** 16 **mp**

Ref Ibid.
D_x 3.90 D_m 3.82 **SS/FOM** F_{30}=11(.014,194)

$\varepsilon\alpha$ 1.750(3) $\eta\omega\beta$ $\varepsilon\gamma$ 1.759(3) **Sign** + **2V** 51(2)
Ref Ibid.

Color Brown to pinkish-brown
Specimen from Hagendorf-Sud pegmatite, Bavaria, Germany. Chemical analysis (wt.%): P_2O_5 31.84, Fe_2O_3 0.38, FeO 37.20, MnO 24.77, MgO 0.42, ZnO 0.42, CaO 0.04, Li_2O 0.19, Na_2O 0.06, H_2O 4.48, F tr: $(Fe_{0.59}Mn_{0.40}Mg_{0.01})_2PO_4(OH)$. $Pb(NO_3)_2$ used as internal standard. Triplite group. PSC: mP144. To replace 5-612.

d(Å)	I/I$_0$	hkl	2θ(°)
5.841	<5	200	19.092
4.670	5	002	23.942
4.382	10	$\bar{2}21$	25.542
3.735	5	310	30.062
3.649	25	221	30.788
3.523	<5	$\bar{2}31$	31.918
3.381	20	122	33.297
3.306	10	040	34.075
3.191	80	202	35.343
3.165	5	$\bar{3}22$	35.643
3.098	100	212	36.440
2.929	70	$\bar{4}02$	38.624
2.881	30	$\bar{2}41$	39.293
2.817	60	023	40.224
2.791	15	$\bar{3}32$	40.615
2.698	10	042	42.081
2.640	10	$\bar{2}42$	43.051
2.570	50	$\bar{3}41$	44.284

Table 1 *(continued)*

d(Å)	I/Io	h k *l*	2θ(°)
2.451	10	$\bar{2}04$	46.558
2.394	5	421	47.734
2.333	10	004	49.063
2.293	20	223	49.977
2.271	5	$\bar{5}22$	50.495
2.220	<5	$\bar{3}24$	51.740
2.192	5	$\bar{4}42$	52.451
2.146	10	061	53.664
2.134	10	114	53.990
2.0629	15	034	56.011
2.0419	10	$\bar{6}02$	56.639
2.0171	<5	$\bar{6}12$	57.399
2.0120	5	$\bar{1}62$	57.559
1.9689	10	$\bar{2}44$	58.941
1.9650	15	204	59.070
1.9491	15	$\bar{6}21$	59.600
1.9190	5	360	60.632
1.8520	<5	$\bar{6}31$	63.071
1.8269	10	$\bar{4}44$	64.040
1.7981	10	270	65.191
1.7930	20	$\bar{4}25$	65.400
1.7821	5	$\bar{6}30$	65.850

Table 2
Chemical Composition of Wolfeite from Hagendorf-Sud

	1	2	3	4
P_2O_5	31.72	31.84	31.75	15.998
Fe_2O_3	—	0.38	—	—
FeO	37.79	37.20	37.53	18.683
MnO	25.51	24.77	25.16	12.676
MgO	—	0.42	0.43	0.393
ZnO	—	0.42	0.43	0.197
CaO	—	0.04	0.04	0.018
Li_2O	—	0.19	—	—
Na_2O	1.18	0.06	0.06	0.072
H_2O	4.03	4.48	4.60	(16.012)
F	0.20	tr	—	—
Total	100.43	99.80	100.00	
O = F	- 0.08			
Total	100.35			

1. Strunz *et al.* (1975)
2. This work; Analyst: J.-M. Speetjens
3. "Z" recalculated to 100% assuming that 0.19 % Li_2O derive from triphylite (0.92 % P_2O_5; 0.65 % FeO; 0.27 % MnO in Fransolet *et al.* 1984) and that 0.38 % Fe_2O_3 are hematite as impurities.
4. Cation numbers on the basis of 144 cationic charges in the cell; the calculated OH-content is given in parentheses.

Other Mineralogical Data

Measured density values determined on two grains by the sink-float method in diluted Clerici solution are 3.82 (2)g/cm^3. Optical properties are: biaxial positive, 2V = 51 (2)° by the method of Mallard, $\alpha \simeq \beta$ = 1.750(3) and γ = 1.759(3), strong r > v dispersion.

A wet chemical analysis was performed on the purified material used for the X-ray measurements. The results are listed in Table 2. Cation numbers were calculated on the basis of 144 cationic charges in the unit cell. The balance of the cationic charges implies that 16.012 OH are required, and that 0.57 wt.% H_2O^- (not involved in the crystal structure) has been determined in excess by the Penfield method. Thus, according to the crystallographic data, Z = 16, and the calculated density is 3.88 g/cm^3.

Taking into account $\bar{n}$ = 1.753 and the actual water content, 4.03 wt.% H_2O, a comparison of the physical specific refractivity *Kp* with the chemical specific refractivity *Kc*, using the revised Gladstone-Dale constant for P_2O_5 of 0.183 (Mandarino, 1981), gives a compatibility index 1 - (*Kp*/*Kc*) of -0.012. The data occupies the superior category on the scale proposed by Mandarino (1981).

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References

Appleman, D. E. & Evans, H. T. , Jr. (1973). Indexing and Least-Squares Refinement of Powder Diffraction Data. Report PB216188. U. S. Dept. of Commerce, National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22151.

Fransolet, A.-M., Antenucci, D., Speetjens, J.-M. & Tarte, P. (1984). *Mineral. Mag. 48*, 373-381.

Frondel, C. (1949). *Am. Mineral. 34*, 692-705.

Mandarina, J. A. (1981). *Can. Mineral. 19*, 441-450.

Strunz, H., Foster, A. & Tennyson, Ch. (1975). *Der Aufschluss, Sonderband 26*, 117-189.

Waldrop, L. (1970). *Z. Kristallogr., Kristallgeom., Kristallphys., Kristallchem. 131*, 1-20.

Yvon, K., Jeitschko, W. & Parthe, E. (1977). *J. Appl. Crystallogr. 10*, 73-74.

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