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LITHIUM-BEARING FLUOR-ARFVEDSONITE FROM HURRICANE MOUNTAIN, NEW HAMPSHIRE: A CRYSTAL-CHEMICAL STUDY

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ABSTRACT

The structures of two crystals of Li-bearing fluor-arfvedsonite (1) $(K_{0.32}Na_{0.68})Na_2(Li_{0.48}Fe^{2+}_{2.83}Mn^{2+}_{0.10}Zn_{0.06}Fe^{3+}_{1.46}Ti_{0.07})(Si_{7.88}Al_{0.12})O_{22}[F_{1.15}(OH)_{0.85}]$ and (2) $(K_{0.25}Na_{0.75})Na_2(Li_{0.48}Fe^{2+}_{2.84}Mn^{2+}_{0.11}Zn_{0.05}Fe^{3+}_{1.45}Ti_{0.07})(Si_{7.89}Al_{0.11})O_{22}[F_{1.35}(OH)_{0.65}]$ from a granitic pegmatite, Hurricane Mountain, New Hampshire, have been refined to R indices of 1.5(1.6)% based on 1380(1387) reflections measured with Mo$K\alpha$ X-radiation. The unit cell parameters are (1) a 9.838(4), b 17.991(6), c 5.315(2) Å, β 103.78(3)°, V 913.7 Å^3 and (2) a 9.832(3), b 17.990(7), c 5.316(3) Å, β 103.79(3)°, V 913.2 Å^3. Site-scattering refinement shows Li to be completely ordered at the $M(3)$ site in these crystals. The amphibole composition is intermediate between fluor-arfvedsonite and fluor-ferro-leakeite with a small component (~10%) of fluor-ferro-ferri-nyböite. These amphibole crystals project into miarolitic cavities in a pegmatitic phase of a riebeckite granite. The early-crystallizing amphibole is close to fluor-ferro-leakeite in composition, but becomes progressively depleted in Li and F as crystals project out into miarolitic cavities; the final amphibole to crystallize is a fibrous Li-poor riebeckite. Li plays a significant role in late-stage fractionation involving the crystallization of alkali amphibole in peralkaline granitic environments.

Keywords: amphibole, fluor-arfvedsonite, lithium, crystal structure, cation order, electron-microprobe analysis, Hurricane Mountain, New Hampshire.

SOMMAIRE

Nous avons affiné la structure de deux cristaux de fluor-arfvedsonite enrichis en Li, de composition (1) $(K_{0.32}Na_{0.68})Na_2(Li_{0.48}Fe^{2+}_{2.83}Mn^{2+}_{0.10}Zn_{0.06}Fe^{3+}_{1.46}Ti_{0.07})(Si_{7.88}Al_{0.12})O_{22}[F_{1.15}(OH)_{0.85}]$ et (2) $(K_{0.25}Na_{0.75})Na_2(Li_{0.48}Fe^{2+}_{2.84}Mn^{2+}_{0.11}Zn_{0.05}Fe^{3+}_{1.45}Ti_{0.07})(Si_{7.89}Al_{0.11})O_{22}[F_{1.35}(OH)_{0.65}]$, provenant d'une pegmatite granitique au mont Hurricane, au New Hampshire, jusqu'à un résidu R de 1.5(1.6)% en utilisant 1380(1387) reflexions mesurées avec rayonnement Mo$K\alpha$. Les paramètres du réseau sont (1) a 9.838(4), b 17.991(6), c 5.315(2) Å, β 103.78(3)°, V 913.7 Å^3 et (2) a 9.832(3), b 17.990(7), c 5.316(3) Å, β 103.79(3)°, V 913.2 Å^3. L'affinement de l'occupation des sites montre que le Li est complètement ordonné sur le site $M(3)$ dans ces cristaux. La composition de ces amphiboles est intermédiaire entre les pôles fluor-arfvedsonite et fluor-ferro-leakeïte; elles contiennent une faible proportion (~10%) de fluor-ferro-ferri-nyböite. Ces cristaux d'amphibole tapissent les parois de cavités miarolitiques dans un faciès pegmatitique d'un granite à riebeckite. La composition de l'amphibole précoce est proche du pôle fluor-ferro-leakeïte, mais elle devient progressivement appauvrie en Li et en F à mesure que le cristal avance vers le centre de la cavité. L'amphibole formée en dernier lieu est une riebeckite fibreuse à faible teneur en Li. Le Li joue un rôle important dans les stades ultimes du fractionnement tardif dans les systèmes granitiques hyperalcalins.

(Traduit par la Rédaction)

Mots-clés: amphibole, fluor-arfvedsonite, lithium, affinement de la structure, degré d'ordre des cations, analyse à la microsonde électronique, mont Hurricane, New Hampshire.

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INTRODUCTION

Lithium can be an important component in alkali amphiboles from peralkaline granites. This has been known for many years (*e.g.*, Borley 1963), but was generally ignored owing to the impossibility of determining the concentration of Li by electron-microprobe analysis. However, improved crystallographic (Hawthorne & Grice 1990), ion-microprobe (Ottolini *et al.* 1993) and LA–ICP–MS (Ludden *et al.* 1995) techniques of analysis have allowed determination of the role of Li in alkali amphiboles (Hawthorne *et al.* 1992, 1993, 1994). Li enters the alkali amphibole (arfvedsonite) structure *via* the substitution

$$^{M(3)}\mathrm{Li} + \mathrm{Fe}^{3+} \rightarrow {}^{M(3)}\mathrm{Fe}^{2+} + \mathrm{Fe}^{2+}$$

giving rise to the end-member amphibole fluor-ferro-leakeite, $\mathrm{NaNa_2Fe^{2+}_2Fe^{3+}_2LiSi_8O_{22}F_2}$ (Hawthorne *et al.* 1996a). Amphibole intermediate between arfvedsonite, fluor-arfvedsonite and various leakeite-related species is probably fairly common in peralkaline granites. Here we report the structural characterization of a Li-bearing amphibole intermediate between arfvedsonite and fluor-ferro-leakeite from granitic pegmatites associated with the Conway Granite, New Hampshire.

EXPERIMENTAL

At Hurricane Mountain, Carroll County, New Hampshire, miarolitic granitic pegmatites occur within an arcuate mass of riebeckite granite that is enclosed by the Conway Granite of the White Mountain series (Creasey & Eby 1993). Within these pegmatites are long (>7 cm) prisms of alkali amphibole, a detailed description of which is given by Foord *et al.* (1996).

TABLE 2. FINAL POSITIONAL PARAMETERS AND EQUIVALENT ISOTROPIC DISPLACEMENT PARAMETERS (Å²) FOR Li-BEARING FLUOR-ARFVEDSONITE* FROM HURRICANE MOUNTAIN

	x	*y*	*z*	$B_{\mathrm{equiv.}}$
O(1)	0.1110	0.0909	0.2071	0.59
	0.1106	0.0911	0.2072	0.65
O(2)	0.1199	0.1718	0.7354	0.62
	0.1199	0.1721	0.7353	0.66
O(3)	0.1122	0	0.7078	1.11
	0.1125	0	0.7081	1.12
O(4)	0.3647	0.2490	0.8009	0.82
	0.3649	0.2489	0.8008	0.86
O(5)	0.3490	0.1274	0.0797	0.77
	0.3489	0.1276	0.0811	0.81
O(6)	0.3407	0.1200	0.5787	0.70
	0.3407	0.1201	0.5784	0.77
O(7)	0.3329	0	0.2987	0.85
	0.3328	0	0.2995	0.90
T(1)	0.2787	0.0858	0.2904	0.41
	0.2787	0.0859	0.2907	0.46
T(2)	0.2894	0.1706	0.8006	0.42
	0.2894	0.1706	0.8010	0.47
M(1)	0	0.08996	1/2	0.58
	0	0.08992	1/2	0.61
M(2)	0	0.18220	0	0.50
	0	0.18222	0	0.54
M(3)	0	0	0	0.61
	0	0	0	0.60
M(4)	0	0.27752	1/2	1.20
	0	0.27767	1/2	1.19
A	0	1/2	0	1.71
	0	1/2	0	1.19
A(*m*)	0.0510	1/2	0.1070	2.30
	0.0468	1/2	0.1028	2.93
A(2)	0	0.4870	0	2.25
	–	–	–	–

* Upper values are for H(1), lower values are for H(2); standard deviations are ≤1 in the last digit

TABLE 1. MISCELLANEOUS INFORMATION FOR Li-BEARING FLUOR-ARFVEDSONITE FROM HURRICANE MOUNTAIN, NEW HAMPSHIRE

	H(1)	H(2)		H(1)	H(2)
Space group	*C*2/*m*	*C*2/*m*			
a (Å)	9.838(4)	9.832(3)	Total Ref.	1380	1387
b (Å)	17.991(6)	17.990(7)	[*F*>5σ(*F*)]	824	908
c (Å)	5.315(2)	5.316(3)	Final *R*(all)	4.67%	3.47%
β (°)	103.78(3)	103.79(3)	Final *R*(obs)	1.48%	1.58%
V (Å³)	913.7	913.2	SEQ	702	703

Cell contents:

H(1): $\mathrm{2[(K_{0.32}Na_{0.68})Na_2(Li_{0.48}Fe^{2+}_{2.83}Mn^{2+}_{0.10}Zn_{0.06}Fe^{3+}_{1.46}Ti_{0.07})(Si_{7.88}Al_{0.12})O_{22}(F_{1.14}\{OH\}_{0.86})}$

H(2): $\mathrm{2[(K_{0.25}Na_{0.75})Na_2(Li_{0.48}Fe^{2+}_{2.84}Mn^{2+}_{0.11}Zn_{0.05}Fe^{3+}_{1.46}Ti_{0.07})(Si_{7.89}Al_{0.11})O_{22}(F_{1.36}\{OH\}_{0.65})}$

$R = \Sigma(|F_o|-|F_c|)/\Sigma|F_o|$

* SEQ = sequence number in Pavia amphibole database

Two crystals were selected for structure study, one fragment from the core and the other from the edge of a large crystal embedded in the granitic pegmatite. The crystals show no visible zoning or inclusions, and have uniform extinction in cross-polarized light. Experimental details for the measurement of unit-cell dimensions and X-ray intensity data are as described by Hawthorne *et al.* (1993). The crystal structures were refined as described by Oberti *et al.* (1992). Unit-cell data, *R* values and other information pertinent to data collection and refinement are listed in Table 1, final atomic coordinates and equivalent isotropic-displacement factors are given in Table 2, refined site-scattering values are given in Table 3, and selected interatomic distances and angles are listed in Table 4. Structure-factor tables may be obtained from The Depository of Unpublished Data, CISTI, National Research Council, Ottawa, Ontario K1A 0S2.

Subsequent to the crystallographic work, the crystals used in the collection of the intensity data were

TABLE 3. REFINED* SITE-SCATTERING VALUES (ELECTRONS PER FORMULA UNIT) AND EQUIVALENT ELECTRONS FROM THE UNIT FORMULAE OF TABLE 5

	H(1)	H(2)
M(1)	51.6	51.6
M(2)	52.2	52.0
M(3)	15.0	14.8
Σ[*M*(1),*M*(2),*M*(3)]	118.8	118.4
EMP [*M*(1),*M*(2),*M*(3)]	118.8	118.6
M(4)+*M*(4′)	22.0	22.0
**MP *M*(4)	22.0	22.0
A	12.5	12.1
**MP *A*	13.5	13.0

* standard deviations are ≤4 in the last digit;
** MP = Electron-and ion-microprobe values.

mounted, polished and analyzed by electron- and ion-microprobe techniques following the procedures described by Oberti *et al.* (1992) and Ottolini *et al.* (1993). The results are given in Table 5, together with the unit formulae calculated on the basis of 24 (O,OH,F) with OH + F = 2 *apfu* (atoms per formula unit) and the ratio of Fe^{3+}/Fe^{2+} derived from the crystal-structure refinement.

TABLE 4. SELECTED INTERATOMIC DISTANCES (Å) AND ANGLES (°) FOR Li-BEARING FLUOR-ARFVEDSONITE FROM HURRICANE MOUNTAIN

	H(1)	H(2)		H(1)	H(2)
T(1)–O(1)	1.606	1.609	*M*(2)–O(1) x2	2.128	2.124
T(1)–O(5)	1.631	1.628	*M*(2)–O(2) x2	2.048	2.048
T(1)–O(6)	1.629	1.626	*M*(2)–O(4) x2	1.937	1.937
T(1)–O(7)	1.631	1.631	<*M*(2)–O>	2.038	2.036
<*T*(1)–O>	1.624	1.623			
			M(3)–O(1) x4	2.122	2.124
T(2)–O(2)	1.620	1.619	*M*(3)–O(3) x2	2.109	2.109
T(2)–O(4)	1.594	1.592	<*M*(3)–O>	2.118	2.119
T(2)–O(5)	1.654	1.656			
T(2)–O(6)	1.660	1.663	*M*(4)–O(2) x2	2.424	2.420
<*T*(2)–O>	1.632	1.633	*M*(4)–O(4) x2	2.360	2.358
			M(4)–O(5) x2	2.923	2.914
M(1)–O(1) x2	2.104	2.100	*M*(4)–O(6) x2	2.519	2.514
M(1)–O(2) x2	2.103	2.106	<*M*(4)–O>	2.557	2.552
M(1)–O(3) x2	2.116	2.117			
<*M*(1)–O>	2.108	2.108	*A*–O(5) x4	2.817	2.823
			A–O(6) x4	3.234	3.236
A(*m*)–O(5) x2	3.013	2.996	*A*–O(7) x2	2.542	2.546
A(*m*)–O(5) x2	2.773	2.778	<*A*–O>	2.929	2.933
A(*m*)–O(6) x2	2.777	2.814			
A(*m*)–O(7)	2.580	2.573	O(5)–O(6)–O(5)	173.3	173.3
A(*m*)–O(7)	3.085	3.130	*T*(1)–O(5)–*T*(2)	135.5	135.6
A(*m*)–O(7)	2.676	2.665	*T*(1)–O(6)–*T*(2)	140.0	140.1
<*A*(*m*)–O>	2.830	2.838	*T*(1)–O(7)–*T*(1)	142.4	142.6

* standard deviations are ≤1 in the last digit

TABLE 5. CHEMICAL COMPOSITIONS* AND UNIT FORMULAE OF Li-BEARING FLUOR-ARFVEDSONITE FROM HURRICANE MOUNTAIN

	H(1)	H(2)		H(1)	H(2)
SiO_2	50.31	49.87	Si	7.883	7.876
Al_2O_3	0.63	0.61	Al	0.116	0.114
TiO_2	0.57	0.60	Sum T	7.999	7.990
Fe_2O_3	12.00	12.00			
FeO	22.14	21.61	Ti	0.067	0.071
MnO	0.77	0.84	Fe^{3+}	1.415	1.426
ZnO	0.49	0.44	Fe^{2+}	2.901	2.854
Na_2O	8.82	8.98	Mn^{2+}	0.102	0.112
K_2O	1.58	1.56	Zn	0.057	0.051
Li_2O	0.76	0.76	Li	0.479	0.483
F	2.31	2.71	Sum C	5.021	4.997
H_2O	(0.82)	(0.61)			
O=F	−0.97	−1.14	^{B}Na	2.000	2.000
	100.26	99.48			
			^{A}Na	0.680	0.750
			K	0.316	0.314
				0.996	1.064
			F	1.145	1.354
			OH	0.855	0.646

* Mg, Ca, Cr, Ni, V not detected. The proportion of constituents is expressed in wt.%.

SITE POPULATIONS

The T sites

There is only a small amount of Al (~0.12 *apfu*) in these amphiboles. Recalculation of the unit formulae (Table 5) indicates that the Al must be assigned to the *T* sites, as the Si content is less than 8.0 *apfu*, and there is no indication of transition metals at either the *T*(1) or *T*(2) sites. The mean bond-lengths (Table 5) at the *T*(1) and *T*(2) sites indicate that *T*(2) is occupied entirely by Si, and that the [4]-coordinated Al occupies the *T*(1) site, as is normally the case in amphiboles (Oberti *et al.* 1995).

The M(1,2,3) sites

There are six chemical species to be distributed over these three *M* sites: Li, Ti^{4+}, Mn^{2+}, Fe^{3+}, Fe^{2+} and Zn. With regard to scattering power, these species can be divided into two groups, Li ($Z = 3$) and Fe* ($Z \approx 26$); note that the amount of Ti ($Z = 22$) and Zn ($Z = 30$) are very similar (and small), and hence their approximation by Fe ($Z = 26$) will not introduce significant error into the refinement process.

Li: The refined scattering values (Table 3) indicate the following mean atomic numbers, Z^{M}, at each site: $Z^{M(1)} \approx Z^{M(2)} \approx 26$, $Z^{M(3)} \approx 15$. This shows directly that Li is completely ordered at the *M*(3) site, as has been found in previous studies (Hawthorne *et al.* 1992, 1993, 1994). The amount of Li in these crystals as determined by SIMS (Table 5) [*i.e.*, $M(3)$ = 0.48 Li + 0.52 Fe*] indicates an effective scattering power at the $M(3)$ site of $0.48 \times 3 + 0.52 \times 26 = 15.0$ *epfu*, in very close agreement with the refined $M(3)$ site-scattering values (Table 3).

Ti^{4+}: In natural amphiboles, $^{[6]}Ti^{4+}$ has been shown to occur at the $M(1)$ site, and is considered to occur most commonly at the $M(2)$ site. Oberti *et al.* (1992) showed that Ti^{4+} enters $M(1)$ *via* the substitution $^{M(1)}Ti^{4+} + 2^{O(3)}O^{2-} \rightarrow {}^{M(1)}(Mg,Fe^{2+}) + 2^{O(3)}(OH)^-$. This substitution is associated with a significant shortening of the $M(1)$–$O(3)$ bond. In the two amphiboles examined here, $M(1)$–$O(3)$ is longer than the other $M(1)$–O bond-lengths (Table 4), indicating that Ti^{4+} does not occur at $M(1)$. Thus we assign all Ti^{4+} to the $M(2)$ site.

Zn: The scattering power of Ti ($Z = 22$) is slightly less than that of Fe^{2+} ($Z = 26$) and yet the refined scattering values at $M(2)$ are slightly larger than the values at $M(1)$. This can only be the case if Zn ($Z = 30$) occurs at $M(2)$ to compensate for the effect of Ti^{4+} at $M(2)$. Hence we assign Zn to the $M(2)$ site.

Fe^{2+} and Fe^{3+}: We are left with Fe^{2+}, Fe^{3+} and Mn^{2+} to assign over the $M(1)$, $M(2)$ and $M(3)$ sites. As these species scatter X-rays in an almost identical fashion, we must rely on stereochemical relationships to assign these species. First, we will consider the species $(Fe,Mn)^{2+}$ and Fe^{3+}, as Fe^{2+} (r = 0.780 Å) and Mn^{2+} (r = 0.830 Å) are more similar to each other than to Fe^{3+} (r = 0.645 Å) in both size and formal charge. As <$M(2)$–O> is much shorter than <$M(1)$–O> and <$M(3)$–O> (Table 4), Fe^{3+} must be strongly ordered at $M(2)$. Following the quantitative arguments of Hawthorne *et al.* (1993), the Fe^{3+} and Fe^{2+} contents of the $M(2)$ site were calculated and used in the derivation of the final unit formulae (Table 5). Mn^{2+} was assigned to $M(3)$ on the basis of the observed mean bond-lengths at the $M(1)$ and $M(3)$ sites.

M(4) site

The site-scattering values (Table 3) are compatible with complete occupancy of the $M(4)$ site by Na, as indicated by the unit formulae determined by electron- and ion-microprobe analysis (Table 5).

A site

The *A* site in each amphibole is occupied by both Na and K. The unit formulae (Table 5) indicate somewhat more scattering power than the results of the site-scattering refinement. This presumably results from the problems of adequately modeling the scattering within the *A* cavity (see Hawthorne et al. 1996b for details) and problems of adequately normalizing the microprobe-determined chemical composition to produce a unit formula.

TABLE 6. SITE POPULATIONS FOR Li-BEARING FLUOR-ARFVEDSONITE FROM HURRICANE MOUNTAIN

Site	Species	H(1)	H(2)
T(1)	Si	3.88	3.89
	Al	0.12	0.11
T(2)	Si	4.00	4.00
M(1)	Fe^{2+}	2.00	2.00
M(2)	Ti^{4+}	0.07	0.07
	Zn	0.06	0.05
	Fe^{3+}	1.46	1.45
	Fe^{2+}	0.41	0.43
M(3)	Li	0.48	0.48
	Fe^{2+}	0.42	0.41
	Mn^{2+}	0.10	0.11
M(4)	Na	2.00	2.00
A	Na	0.68	0.75
	K	0.32	0.25
O(3)	F	1.15	1.35
	*OH	(0.85)	(0.65)

* Calculated as 2 – F.

Final site-populations for each crystal are given in Table 6. Comparison of the aggregate site-scattering values of the *A*-, *B*- and *C*-group cations from site-scattering refinement and *via* renormalization of the microprobe determination is made in Table 3.

CHEMICAL COMPOSITION

Crystals H(1) and H(2) each have 0.48 *Li apfu* ordered at the $M(3)$ site. Taking fluor-arfvedsonite, $NaNa_2(Fe^{2+}_4Fe^{3+})Si_8O_{22}F_2$ as the root (additive) composition, Li enters the amphibole structure *via* the substitution $^{M(3)}Li + Fe^{3+} \rightarrow {}^{M(3)}Fe^{2+} + Fe^{2+}$; stereochemical aspects of this substitution were discussed by Hawthorne *et al.* (1993, 1994). Both crystals of the present study lie almost half-way between fluor-arfvedsonite and fluor-ferro-leakeite, $NaNa_2(Fe^{2+}_2Fe^{3+}_2Li)Si_8O_{22}F_2$ (Hawthorne *et al.* 1996a), and the small amount of [4]-coordinated Al indicates a small (~10%) component of fluor-ferro-ferri-nyböite. Both crystals are thus Li-bearing fluor-arfvedsonite. The compositions of H(1) and H(2) fall on the compositional trends of alkali amphiboles from the Questa caldera, New Mexico (Hawthorne *et al.*, 1993, Fig. 7). They are far less manganiferous, reflecting differences in bulk-rock composition (Czamanske & Dillet 1988, Foord *et al.* 1996), but it is notable that both sets of amphiboles contain significant Zn (ZnO up to 0.90 and 0.49 wt% at Questa and Hurricane Mountain, respectively).

PARAGENESIS

Details of the paragenesis of this amphibole are given by Foord *et al.* (1996). The earliest amphibole to crystallize is Li-bearing fluor-arfvedsonite, represented by crystals H(1) and H(2). The amphibole composition becomes poorer in Li and F along the length of the large crystals as they project out into the miarolitic cavities. Later fibrous Li-poor riebeckite partly fills some of the miarolitic cavities, forming casts over quartz and feldspar. Thus, we can identify the following paragenetic trend in amphibole composition:

Li-rich fluor-arfvedsonite → (ferro-fluor-leakeite) → Li-bearing arfvedsonite → late-stage riebeckite. Presumably the amphibole depletes the late hydrothermal fluids in Li and F, the latest phases becoming OH-rich and fibrous. It may be of significance that similar trends in both composition and morphology occur in tourmaline from miarolitic cavities in peraluminous granitic pegmatites.

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