

The Canadian Mineralogist
Vol. 39, pp. 171-177 (2001)

FERRIAN WINCHITE FROM THE ILMEN MOUNTAINS, SOUTHERN URALS, RUSSIA, AND SOME PROBLEMS WITH THE CURRENT SCHEME FOR AMPHIBOLE NOMENCLATURE

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ABSTRACT

The crystal structure of ferrian winchite ($\square_{0.87}\text{K}_{0.13}\Sigma_{1.00}(\text{Ca}_{0.65}\text{Na}_{1.34})\Sigma_{1.99}(\text{Mg}_{2.78}\text{Fe}^{2+}_{0.12}\text{Mn}_{0.07}\text{Ti}_{0.01})\Sigma_{2.98}(\text{Fe}^{2+}_{0.88}\text{Fe}^{3+}_{0.92}\text{Al}_{0.20})\Sigma_{2.00}(\text{Si}_{7.88}\text{Al}_{0.12})\Sigma_{8.00}\text{O}_{22}[(\text{OH})_{1.77}\text{F}_{0.23}]\Sigma_{2.00}$, a 9.8339(2), b 18.0357(2), c 5.2974(1) Å, β 104.195(1)°, V 910.9(2) Å³, $C2/m$, $Z = 2$, $D(\text{calc}) = 3.12 \text{ g/cm}^3$, has been refined using the Rietveld method to the following values: $R_p = 4.1$ and $R_F = 3.6\%$, $s = 2.74$. The $\text{Fe}^{3+}/(\text{Fe}^{2+} + \text{Fe}^{3+})$ value, 0.48, was determined by Mössbauer spectroscopy. Site populations were assigned on the basis of the refined site-scattering values at the A-, B- and C-group sites. ²⁷Al was assigned to the T(1) site of the tetrahedral double chain on the basis of the observed $\langle T\text{--O} \rangle$ distances. In the structure of ferrian winchite, all Fe^{3+} occurs at the M(2) site, in accord with the observed $\langle M(2)\text{--O} \rangle$ distance of 2.05 Å. Fe^{2+} is partitioned between the M(2) and M(3) sites. The high amount of Fe^{3+} (0.92 apfu) at the M(2) site focuses attention on some defects of the current system of amphibole nomenclature.

Keywords: ferrian winchite, amphibole, crystal structure, Rietveld refinement, Mössbauer spectroscopy, chemical analysis.

SOMMAIRE

Nous avons affiné la structure cristalline d'un échantillon de winchite riche en fer ferrique ($\square_{0.87}\text{K}_{0.13}\Sigma_{1.00}(\text{Ca}_{0.65}\text{Na}_{1.34})\Sigma_{1.99}(\text{Mg}_{2.78}\text{Fe}^{2+}_{0.12}\text{Mn}_{0.07}\text{Ti}_{0.01})\Sigma_{2.98}(\text{Fe}^{2+}_{0.88}\text{Fe}^{3+}_{0.92}\text{Al}_{0.20})\Sigma_{2.00}(\text{Si}_{7.88}\text{Al}_{0.12})\Sigma_{8.00}\text{O}_{22}[(\text{OH})_{1.77}\text{F}_{0.23}]\Sigma_{2.00}$, a 9.8339(2), b 18.0357(2), c 5.2974(1) Å, β 104.195(1)°, V 910.9(2) Å³, $C2/m$, $Z = 2$, $D(\text{calc}) = 3.12 \text{ g/cm}^3$, par la méthode de Rietveld, ce qui a donné les valeurs suivantes: $R_p = 4.1$ et $R_F = 3.6\%$, $s = 2.74$. Le rapport $\text{Fe}^{3+}/(\text{Fe}^{2+} + \text{Fe}^{3+})$, 0.48, a été déterminé par spectroscopie de Mössbauer. Nous avons assigné l'occupation des sites selon les résultats d'un affinement des valeurs de dispersion aux sites A, B et C. L'aluminium tétracoordonné a été attribué au site T(1) de la chaîne double de tétraèdres en fonction des distances $\langle T\text{--O} \rangle$ observées. Dans cette structure, tout le Fe^{3+} occupe le site M(2), en accord avec la distance $\langle M(2)\text{--O} \rangle$ observée, 2.05 Å. Le Fe^{2+} est réparti entre les sites M(2) et M(3). La proportion élevée de Fe^{3+} sur le site M(2) (0.92 atomes par unité formulaire) soulève des questions à propos du système de classification des amphiboles utilisé actuellement.

(Traduit par la Rédaction)

Mots-clés: winchite riche en fer ferrique, amphibole, structure cristalline, affinement de Rietveld, spectroscopie de Mössbauer, données chimiques.

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INTRODUCTION

Until now, winchite, ideally $(\text{CaNa}) \text{Mg}_4 (\text{Al}, \text{Fe}^{3+}) \text{Si}_8 \text{O}_{22} (\text{OH})_2$, has been insufficiently studied, and there is a lack of information on this species. Chemical data are very contradictory (Bilgrami 1955, Nayak & Leake 1975). Wylie & Huggins (1980) reviewed previously published data and concluded that many previously identified samples of "winchite" are not winchite at all: for example, "of the seven analyses of 'winchite' listed by Nayak & Leake (1975), only one referred to a winchite". Ferrian winchite, a silica-rich sodic-calcic amphibole, $(\square_{0.87}\text{K}_{0.13})_{\Sigma 1.00} (\text{Ca}_{0.65}\text{Na}_{1.34})_{\Sigma 1.99} (\text{Mg}_{2.78}\text{Fe}^{2+}_{0.12}\text{Mn}_{0.07}\text{Ti}_{0.01})_{\Sigma 2.98} (\text{Fe}^{2+}_{0.88}\text{Fe}^{3+}_{0.92}\text{Al}_{0.20})_{\Sigma 2.00} (\text{Si}_{7.88}\text{Al}_{0.12})_{\Sigma 8.00} \text{O}_{22} [(\text{OH})_{1.77}\text{F}_{0.23}]_{\Sigma 2.00}$, was recently found in a contact zone of the Ilmen alkaline massif, Ilmen Mountains, southern Urals, Russia. From the same locality, potassic-ferrisadanagaite $(\text{K}, \text{Na}) \text{Ca}_2 (\text{Fe}^{2+}, \text{Mg})_3 (\text{Fe}^{3+}, \text{Al})_2 [\text{Si}_5\text{Al}_3\text{O}_{22}] (\text{OH})_2$, a new mineral of the amphibole group, was first described (Bazhenov *et al.* 1999, Sokolova *et al.* 2000a). Details of the geology and mineralogy of the locality are given by Bazhenov *et al.* (1999). In contrast to the chemically homogeneous potassic-ferrisadanagaite, ferrian winchite samples show wide variation in chemical composition. In this paper, we present results of a structure investigation of ferrian winchite by Rietveld refinement and Mössbauer spectroscopy.

EXPERIMENTAL DETAILS

Mössbauer spectroscopy

A sample of ferrian winchite was ground gently in an agate mortar with acetone, and mixed with benzophe-

none to reduce preferred orientation of the crystallites. The Mössbauer-absorber thickness for the sample was 5 mg Fe/cm^2 . The spectrum was collected at room temperature (293 K) on a conventional transmission Mössbauer spectrometer.

The spectrum of ferrian winchite is typical of those for amphiboles, and was fitted to two Voigt doublets corresponding to Fe^{2+} and one Voigt doublet corresponding to Fe^{3+} . Component areas and widths of the doublets were constrained to be equal, and the Lorentzian linewidth of the Voigt lineshape was fixed at the natural value of 0.195 mm/s . For Fe^{2+} (I) and (II), the center shifts are $1.150(5)$ and $1.071(6) \text{ mm/s}$ (relative to $\alpha\text{-Fe}$), the quadrupole splittings are $2.809(5)$ and $2.27(4) \text{ mm/s}$, the Gaussian standard deviations are $0.088(3)$ and $0.19(2) \text{ mm/s}$, and the relative areas are 36 and 16%, respectively. For Fe^{3+} , the center shift is $0.385(5) \text{ mm/s}$ (relative to $\alpha\text{-Fe}$), the quadrupole splitting is $0.478(5) \text{ mm/s}$, the Gaussian standard deviation is $0.082(2) \text{ mm/s}$, and the relative area is 48%.

X-ray powder diffraction

Powder diffraction was used because of the fibrous nature of the crystals of ferrian winchite (Fig. 1). Powder-diffraction data were collected on a focusing STOE-STADIP diffractometer, equipped with a curved Ge (111) primary monochromator that produces monochromatic $\text{MoK}\alpha_1$ radiation ($\lambda = 0.70926 \text{ \AA}$) with a minimum FWHM of 0.08° , according to the procedure of Wölfe (1981). The sample was contained in a quartz capillary, which was rotated to minimize preferred orientation. Diffracted intensities were collected in stepwise-overlapping mode of a linear position-sensitive detector with about 5° acceptance angle and 0.02°

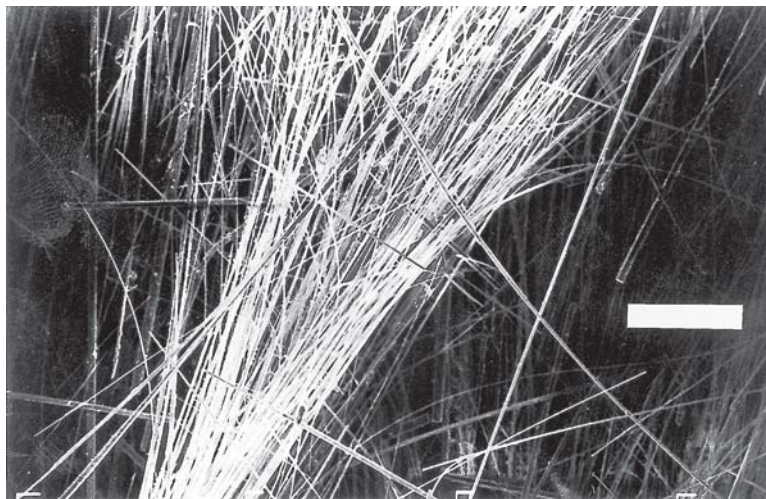


FIG. 1. SEM image of fibrous crystals of ferrian winchite. The scale bar represents $200 \mu\text{m}$.

TABLE 1. CRYSTALLOGRAPHIC DATA FOR FERRIAN WINCHITE

	Ferrian winchite	Quartz
<i>a</i> (Å)	9.8339(2)	4.915(1)
<i>b</i> (Å)	18.0357(2)	
<i>c</i> (Å)	5.2974(1)	5.401(1)
β (°)	104.195(1)	
<i>V</i> (Å ³)	910.9(2)	113.0(4)
<i>Z</i>	2	3
Space group	<i>C2/m</i>	<i>P3₂21</i>
<i>D</i> _{calc} (g/cm ³)	3.12	2.65
2θ (°)-range	2.00–54.98	2.00–54.98
Asymmetry, 2θ (°)	< 30	–
Bragg reflections	1137	126
Refined parameters	65	5
<i>R</i> _p (%)	4.13	4.13
<i>R</i> _{wp} (%)	5.79	5.79
<i>R</i> _{exp} (%)	2.11	2.11
<i>R</i> _B (%)	4.25	7.19
<i>R</i> _c (%)	3.63	7.03
<i>s</i> *	2.74	2.74
<i>DWD</i> **	0.52	0.52
σ_x ***	1.956	1.956
Wt. %	96.50(1)	3.50(1)

s* = *R*_{wp}/*R*_{exp} - expected value of *R*_{wp}*DWD*: Durbin-Watson *d* statistic (Hill & Flack 1987).*** σ_x : multiplier for the E.S.D.'s (Bérar & Lelann 1991)

channel (Wölffel 1983); the yield was 2650 data points in the range $2.00 < 2\theta \leq 54.98^\circ$ (see Table 1).

A Rietveld refinement of the ferrian winchite structure, using the atom coordinates for strontian potassic-chrichterite (Sokolova *et al.* 2000b) as a starting model, was done with the WYRIT 3.3 program (Schneider 1989) using a Pearson VII reflection-profile function. The reflection profiles were fitted over six full-width-at-half-maximum (FWHM) values. Graphical modeling of background was used, and asymmetry was refined for reflections with $2\theta < 30^\circ$. Occupancies of the *A* and *M* sites were refined.

The refinement was complicated by the presence of quartz in the sample. Besides joint instrumental and profile parameters, scale factor and cell parameters were refined for quartz.

RESULTS

Mössbauer spectroscopy

The Fe²⁺ absorption consists of strongly overlapped doublets, and the parameters cannot be determined precisely (Fig. 2). However, the resulting parameters are typical for Fe²⁺ occupying one or more of the *M*(1), *M*(2) and *M*(3) sites in the amphibole structure, but reliable site-distributions for Fe²⁺ cannot be determined owing to line overlap. There is no detectable evidence of Fe²⁺ occupying the *M*(4) site. The Fe³⁺ absorption was

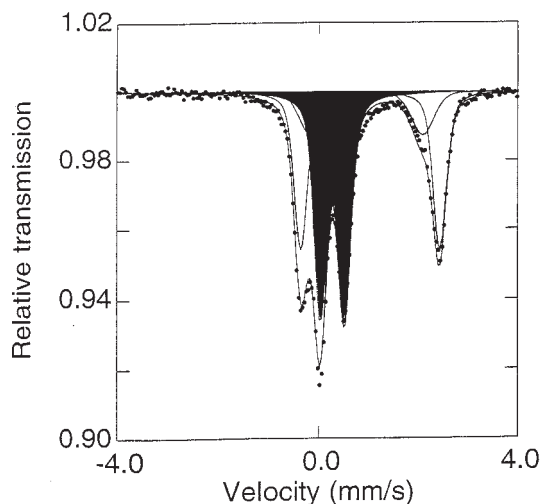


FIG. 2. Room-temperature Mössbauer spectrum of ferrian winchite showing a fit to two Fe²⁺ doublets (unshaded) and one Fe³⁺ doublet (shaded).

fitted to a single quadrupole doublet, and an Fe³⁺/ΣFe ratio of 0.48 was determined from the refined area-ratios.

Rietveld refinement

Final results are characterized by *R*_{wp} = 0.058, *s* = *R*_{wp}/*R*_{exp} (goodness of fit) of 2.74, Durbin-Watson *d*-statistic *DWD* (Hill & Flack 1987) of 0.52. All the estimated standard deviations have been multiplied by $\sigma_x = 1.956$ (Bérar & Lelann 1991) to correct for serial correlation. A quantitative analysis estimated the amount of admixed quartz as 3.5(1) wt.%. The refined powder pattern is shown in Figure 3. Table 2 presents atom coordinates and isotropic-displacement factors, Table 3 lists selected interatomic distances and angles, and Table 4 gives the refined site-scattering values.

DISCUSSION

Site populations

The refined site-scattering values are given in Table 4 in terms of *epfu* (electrons per formula unit). The value for *M*(1) indicates that this site is completely occupied by Mg. The site scattering at *M*(2) is extremely high, relative to the values at the *M*(1) and *M*(3) sites, indicating that the strong scatterers in the unit formula, specifically Fe, are concentrated at *M*(2). Most amphiboles have their higher-valence *C*-group cations ordered at *M*(2); consequently, we assigned Fe²⁺, Fe³⁺, Ti and Al to *M*(2) in accord with the refined site-scattering value. Ferrous iron, Mn and Mg were assigned to the *M*(3) site to produce the refined site-scattering value (Table 4).

TABLE 4. REFINED SITE-SCATTERING VALUES ($epfu$) AND ASSIGNED SITE-POPULATIONS ($apfu$) FOR FERRIAN WINCHITE

	Refined site-scattering	Site-population *	Calculated site-scattering
<i>M</i> (1)	24	2.00 Mg	24
<i>M</i> (2)	49.4	0.92 Fe ³⁺ + 0.87 Fe ²⁺ + 0.20 Al + 0.01 Ti	49.4
<i>M</i> (3)	14.8	0.13 Fe ²⁺ + 0.79 Mg + 0.07 Mn	14.6
<i>M</i> (4)	27.9	1.34 Na ⁺ + 0.66 Ca	27.9
<i>A</i>	2.47	0.13 K	2.47

TABLE 5. CHEMICAL COMPOSITION AND FORMULA UNIT (*apfu*) FOR FERRIAN WINCHITE

Bazhenov (pers. commun.)	This work *
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SiO ₂	54.52	Si	7.78	Si	7.80
TiO ₂	0.08	[⁴ Al]	0.22	[⁴ Al]	0.20
Al ₂ O ₃	2.33	ΣT	8.00	ΣT	8.00
Fe ₂ O ₃	9.33				
FeO	8.08	[⁶ Al]	0.17	[⁶ Al]	0.20
MnO	0.61	Ti	0.01	Ti	0.01
MgO	13.67	Fe ³⁺	1.00	Fe ³⁺	0.92
CaO	4.44	Fe ²⁺	0.96	Fe ²⁺	1.00
Na ₂ O	4.75	Mn	0.07	Mn	0.07
K ₂ O	0.63	Mg	2.91	Mg	2.79
H ₂ O	1.75	ΣC	5.12	ΣC	4.99
F	0.75				
F=O	<u>-0.32</u>	Ca	0.68	Mg	0.29
Total	100.62	Na	1.31	Ca	0.66
		ΣB	1.99	Na	1.05
				ΣB	2.00
		K	0.12		
		ΣA	0.12	K	0.13
				ΣA	0.13
		OH	1.66		
		F	0.34	OH	1.77
				F	0.23

Hawthorne (1983) derived equations between the observed mean bond-lengths and the constituent ionic radii for the $M(1)$, $M(2)$ and $M(3)$ sites in the $C2/m$ amphiboles. Using the site populations of Table 4, the calculated $\langle M-O \rangle$ distances for the $M(1)$, $M(2)$ and $M(3)$ sites are 2.08, 2.06 and 2.08 Å, respectively, in reasonably good agreement with the observed values of 2.08, 2.05 and 2.09 Å (Table 3).

Originally, the sample in question was considered as a possible new species, *i.e.*, “ferriwinchite”. However, both site-refinement results and Mössbauer spectroscopy indicate a lower amount of Fe³⁺ than in the wet-chemical data (Table 5). The unit formula shown in Table 5 (this work) was derived from the results of the site-scattering refinement combined with the Mössbauer results, aspects of the wet-chemical data (Table 5), and stereochemical features of the structure.

In the unit formula of Table 5 (this work), the Ti and Mn contents were assigned from the bulk chemical composition of A.G. Bazhenov. The unit formula recalculated from this latter analysis indicates only K at the A site and Ca and Na* (= Na + Mg) at the *M*(4) site. The refined site-scattering values were interpreted accord-

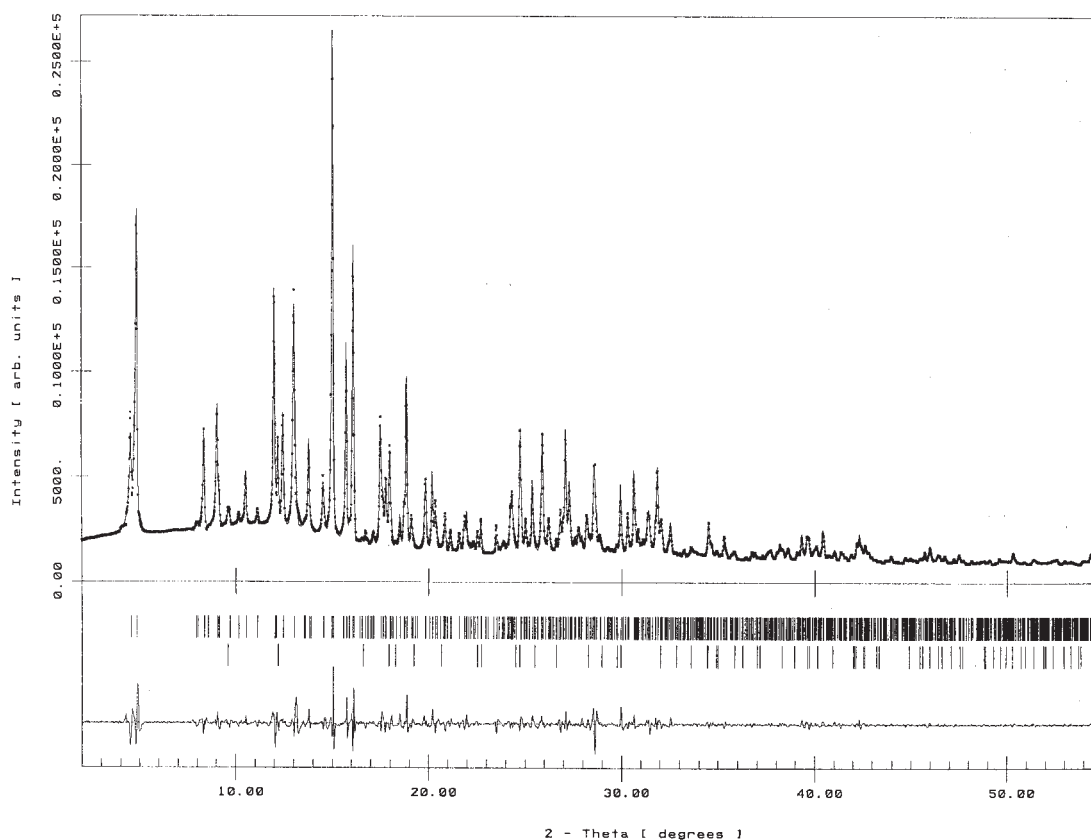


FIG. 3. The X-ray powder-diffraction pattern for ferrian winchite: the observed pattern is shown by the small circles (dots), the calculated pattern is the continuous line, the vertical lines show the positions of all Bragg reflections for ferrian winchite and quartz (below); at the bottom, there is the difference (observed minus calculated) pattern.

ingly, and the resultant cation occupancies (Table 4) are in good accord with the unit formula of Bazhenov reported in Table 5. The C-group cations were derived as the sum of the $M(1,2,3)$ site-populations (Table 4). The $\langle T-O \rangle$ distances indicate that $T(2)$ is occupied totally by Si, and that the $T(1)$ site contains 0.20 Al *pfu* (per formula unit) according to the curve of Hawthorne (1983). The resulting formula was made neutral by assigning the Na^* ($= Na + Mg$) at $M(4)$ accordingly (Table 4). This amphibole is ferrian winchite.

Winchite

Characterization of this particular ferrian winchite raises some interesting points with regard to amphibole classification. The current classification for amphiboles (Leake *et al.* 1997) defines end-member winchite as follows:



This is not an end member as an end member has to have a fixed composition, otherwise it can be resolved into two (or possibly more) components of fixed composition that will be different end-members. Thus the winchite "end-member" defined above is intermediate between



and



These two compositions are fixed and hence should be formally regarded as end members from an algebraic perspective.

There are several amphibole compositions that are formally defined as end members by Leake *et al.* (1997) that have variable composition:

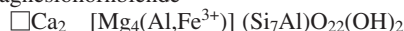
Magnesiosadanagaite



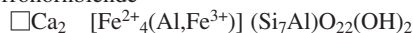
Sadanaite



Magnesiohornblende



Ferrohornblende



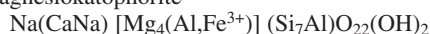
Winchite



Ferrowinchite



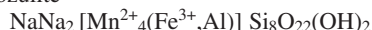
Magnesiokatophorite



Katophorite



Kozulite



All of these compositions are solid solutions between distinct Al and Fe^{3+} end-members.

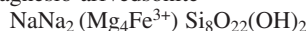
Consider the composition $\square (\text{CaNa}) (\text{Mg}_4\text{Fe}^{3+}) \text{Si}_8\text{O}_{22}(\text{OH})_2$; this has the name winchite under the current scheme of classification. Consider now the composition $\square (\text{Ca}_{0.99}\text{Na}_{1.01}) (\text{Mg}_{3.99}\text{Fe}^{3+}_{1.01}) \text{Si}_8\text{O}_{22}(\text{OH})_2$; according to the current scheme, this will have the name “ferriwinchite” as $\text{Fe}^{3+} > 1.00$ apfu (atoms per formula unit). So now we are left with the question as to what is the end-member composition of “ferriwinchite”. It must have an end-member composition as it is a distinct name; however, the end-member composition cannot be $\square (\text{CaNa}) (\text{Mg}_4\text{Fe}^{3+}) \text{Si}_8\text{O}_{22}(\text{OH})_2$ as this composition has $\text{Fe}^{3+} \neq 1.00$ apfu. Taking the Fe^{3+} content to 2 apfu produces amphiboles that are not of a winchite-like composition. It is apparent that we have an inconsistency in the current nomenclature scheme with regard to this point.

In the sodic amphiboles, it is instructive to consider the following two sets of compositions:

Eckermannite



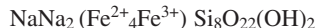
Magnesio-arfvedsonite



Ferro-eckermannite



Arfvedsonite



In these particular cases, the Al and Fe^{3+} end-members are identified appropriately. There seems no reason why kozulite and “aluminokozulite” should not likewise be defined as the corresponding Fe^{3+} and Al end-member compositions, for example.

Of course, the problem arises by trying to define the prefix *ferri* in terms of the amount of Fe^{3+} pfu. In principle, the same problems would arise with Fe^{2+} and Mg

if compositions were defined in terms of apfu of these variables. However, this is not the case for Fe^{2+} and Mg; compositional boundaries between species are defined by ratios between these components; i.e., Mg / (Mg + Fe^{2+}). Thus some end-members contain Mg_3 (e.g., tschermakite) and others contain Mg_5 (e.g., tremolite), and boundaries between Mg-dominant and Fe^{2+} -dominant species occur at Mg / (Mg + Fe^{2+}) = 0.50, not at specific amounts of Mg pfu. Perhaps the use of Al / (Al + Fe^{3+}) = 0.50 as boundaries between “alumino” and “ferri” species is a better option here. However, this implies using a different set of principal reference-axes (i.e., those involving the A-, B-, and C-group sites rather than those involving the A-, B- and T-group sites) for the nomenclature scheme. This issue is clearly a significant problem in the present system of nomenclature.

ACKNOWLEDGEMENTS

We thank A.G. Bazhenov for the sample of ferrian winchite and corresponding chemical data, and L.A. Pautov for the SEM image of winchite fibers. This work was supported by the Russian Fund for Basic Research, grant 00-05-65399 to EVS, and Natural Sciences and Engineering Research Council of Canada grants to FCH.

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