

Ferri-clinoholmquistite, $\text{Li}_2(\text{Fe}^{2+}, \text{Mg})_3\text{Fe}^{3+}\text{Si}_8\text{O}_{22}(\text{OH})_2$, a new $^{\text{B}}\text{Li}$ clinoamphibole from the Pedriza Massif, Sierra de Guadarrama, Spanish Central System

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ABSTRACT

Ferri-clinoholmquistite is a new amphibole species from episyenites in the East Pedriza Massif (Central System, Spain), where it is associated to albite, augite-aegirine, titanite, andradite, magnetite, and apatite. It is black, vitreous, translucent, non-fluorescent, and brittle. It shows gray streak, $H(\text{Mohs}) = 6$, splintery fracture, perfect $\{110\}$ cleavage, (001) parting, $D_{\text{meas}} = 3.19$, and $D_{\text{cal}} = 3.25$. Crystals are prismatic, elongated on [001]. In plane-polarized light, it is strongly pleochroic: $X = \text{yellow green}$, $Y = \text{indigo blue}$, $Z = \text{green blue}$, with absorption $X < Y \leq Z$; $Z = b$, $Y - c = 10(2)^\circ$, $X - a \sim -2^\circ$ (in β obtuse). Ferri-clinoholmquistite is biaxial positive, $\alpha = 1.699(2)$, $\beta = 1.703(2)$, $\gamma = 1.708(2)$, $2V_z(\text{meas}) = 72(7)$, $2V_z(\text{calc}) = 84(6)$, $r < v$. It is monoclinic, space group $C2/m$, $a = 9.472(4)$, $b = 17.844(6)$, $c = 5.276(6)$ Å, $\beta = 101.97(9)^\circ$, $V = 872(1)$ Å³, $Z = 2$. X-ray powder-diffraction pattern data were determined. Analysis by a combination of electron microprobe and flame photometry gives the following formula, calculated assuming $\text{OH} + \text{F} = 2$ and T sites fully occupied by Si: $^{\text{A}}(\text{Na}_{0.43} \text{K}_{0.03})^{\text{B}}(\text{Li}_{1.66} \text{Na}_{0.30} \text{Ca}_{0.04})^{\text{C}}(\text{Fe}_{1.54}^{3+} \text{Fe}_{1.35}^{2+} \text{Mg}_{1.21} \text{Li}_{0.49} \text{Al}_{0.20} \text{Ti}_{0.12}^{4+} \text{Mn}_{0.07}^{2+} \text{Zn}_{0.02})^{\text{T}}(\text{Si}_8)\text{O}_{22}(\text{OH}_{1.58} \text{F}_{0.42})$. From crystallographic refinements the M4 site is split, implying ordering of Li and Na, and within the A cavity, Na occupies the Am position.

INTRODUCTION

Clinoholmquistite, a poorly known amphibole, was characterized by Ginzburg (1965) and Litvin et al. (1975), as summarized by Hawthorne (1983). This work deals with ferri-clinoholmquistite, a new monoclinic amphibole recently approved by the I.M.A. Commission on New Minerals and Mineral Names. The name is based on the composition, in accord with the nomenclature of Leake (1978a, 1978b). Holotype material has been deposited in the Museo Nacional de Ciencias Naturales, Madrid (Spain), under code numbers FERRI-E-1 (grain concentrate) and FERRI-E-2 (powdered sample).

OCCURRENCE

Ferri-clinoholmquistite occurs in hydrothermal episyenitic bodies located in the east of the Pedriza Massif (Sierra de Guadarrama, Central System, Spain). These episyenites were derived from a Li-rich cordierite-bearing porphyritic granite (Villaseca and Pérez Soba 1989). Aqueous non-magmatic fluids of low salinity, at approximately 520 °C and fluid pressures ranging from 60 to 160 MPa (Caballero 1993), leached quartz from the system, causing albitization and the formation of a new mafic-mineral assemblage, including ferri-clinoholmquistite.

The type material was collected in the Arroyo de la Yedra Valley [Spanish Military Map number 509 (19–

20), scale 1:50000, 4th edition (1986)]. The episyenitic body has a tabular shape, from 1 to 2 m thick and several tens of meters in length and with a subvertical dip: In outcrop, the episyenite preserves the porphyritic texture and general appearance of the granite.

In thin section, the episyenite shows a porphyritic texture. Relict zoned plagioclase phenocrysts, largely replaced by hydrothermal albite, float in an unequigranular matrix of xenoblastic albite and fine-grained intergranular aggregates of albite and mafic minerals.

There are four other minor mineral assemblages, which belong to different stages in the evolution of the rock, observed in addition to the prevailing episyenitic mineral association. Their proposed sequence is (1) a relict granite assemblage, with calcic plagioclase cores in albite phenocrysts, partly albitized microcline crystals in the feldspathic matrix, feldspar-armored biotite and zircon microcrystals and (2) the episyenitic assemblage, with albite, augite-aegirine, ferri-clinoholmquistite (7% modal), titanite, andradite, magnetite, and apatite. The mafic association appears as intergranular granoblastic aggregates associated with fine-grained albite or as micro-inclusions in albite. Apatite is restricted largely to the albitized cores of plagioclase phenocrysts. Augite-aegirine pyroxene is a representative of an early episyenitic stage, being partly replaced by clinoholmquistite; (3) an early retrograde as-

TABLE 1. Mean chemical analysis and formula unit ($n = 6$)

SiO ₂	56.15(45)	Si	8.00	Ca	0.04(1)
TiO ₂	1.16(7)			Li	1.66(4)
Al ₂ O ₃	1.19(6)	Ti	0.12(1)	Na	0.30(3)
Fe ₂ O ₃	14.37(73)	Al	0.20(1)	Total B	2.00
FeO	11.29(57)	Fe ³⁺	1.54(7)	Na	0.44(6)
MnO	0.57(5)	Fe ²⁺	1.35(8)	K	0.03(1)
MgO	5.72(12)	Mn ²⁺	0.07(1)	Total A	0.47(6)
ZnO	0.16(7)	Mg	1.21(3)	O	22.00
CaO	0.25(4)	Zn	0.02(1)	OH	1.58(9)
Li ₂ O	3.75(2)	Li	0.49(2)	F	0.42(9)
Na ₂ O	2.66(18)	Total C	5.00		
K ₂ O	0.17(3)				
H ₂ O	1.66(9)				
F	0.93(20)				
F=O	-0.39				
Sum	99.64 wt%				

semblage of microcline and taeniolite, replacing both the albite and the ferri-clinoholmquistite in the intergranular aggregates and also the feldspar-armored biotite crystals. The replacement of fine-grained albite by microcline gives rise to poikiloblastic crystals of microcline containing micro-inclusions of ferri-clinoholmquistite; (4) a second retrograde assemblage, also developed in the intergranular aggregates, composed of quartz replacing the former feldspars, ferro-actinolite replacing the former amphibole, and minor chlorite replacing both the ferri-clinoholmquistite and micas. Similar to the former process, the replacement of albite by quartz may result in the presence of poikiloblastic quartz with micro-inclusions of ferri-clinoholmquistite; (5) a low-temperature retrograde assemblage of sericite, developed in the calcic plagioclase cores, and oxides and argillitic minerals replacing the mafic minerals.

Ferri-clinoholmquistite has two grain-size subpopulations: Crystals in the intergranular aggregates average 0.15(10) mm in length and 0.11(6) mm in width, whereas micro-inclusions in albite, microcline, quartz, and titanite are 0.11(13) mm in length and 0.03(2) mm in width.

PHYSICAL AND OPTICAL PROPERTIES

Ferri-clinoholmquistite is black, translucent, with a gray streak and vitreous luster. It is brittle with a splintery fracture and Mohs hardness of six. The crystals have prismatic habit, elongated up to 0.5 mm along [001]. The most common form is {110}. The {001} pinacoid is much less common, whereas {100} and {010} pinacoids are rare and always only incipiently developed. The observed density, measured by flotation, is 3.19 g/cm³; the calculated density is 3.25 g/cm³. The observed difference may arise from the presence of abundant fluid inclusions.

Orientation, pleochroism, and absorption data were obtained by universal-stage measurements on grain-mount thin-sections. 2V angles were measured on the basis of extinction data on a spindle stage under yellow monochromatic light ($\lambda = 589.2$ nm), following the experimental procedure described by Bloss (1981). Oriented crystals on the spindle stage were used to measure the indices of refraction using the variation method.

TABLE 2. Indexed powder pattern

hkl	I_{obs}	d_{obs} (Å)	d_{calc} (Å)
110	61	8.222	8.223
040	19	4.458	4.461
240	12	3.217	3.213
310	100	3.044	3.043
330	53	2.741	2.741
151	14	2.712	2.708
112	14	2.341	2.342
171	8	2.270	2.268
261	9	2.159	2.159
222	9	2.025	2.024
351	8	2.012	2.012
371	12	1.643	1.642
352	8	1.609	1.610
1,11,0	9	1.598	1.598
422	8	1.546	1.546
223	6	1.492	1.492
243	46	1.433	1.433
642	14	1.392	1.393
173	11	1.374	1.374

Notes: Unit-cell parameters: $a = 9.472(4)$, $b = 17.844(6)$, $c = 5.276(6)$ Å, $\beta = 101.97(9)^\circ$, $V = 872(1)$ Å³.

Ferri-clinoholmquistite has perfect {110} cleavage intersecting at $\sim 56^\circ$ and a (001) parting. It is strongly pleochroic with $X =$ yellow green, $Y =$ indigo blue and $Z =$ green blue, with absorption $X < Y \sim Z$; $X - a \sim -2^\circ$ (in obtuse angle β), $Y - c = 10(2)^\circ$ and $Z = b$. It is biaxial positive with indices of refraction $\alpha = 1.699(2)$, $\beta = 1.703(2)$, and $\gamma = 1.708(2)$; $2V_z = 72(7)^\circ$ and $2V_{z,\text{calc}} = 83(6)^\circ$, dispersion is $v > r$ and shows anomalous indigo and brown interference colors. Calculation of the Gladstone-Dale relationship yielded a compatibility index symbol ($1 - K_p/K_c$) of 0.013 (Mandarino 1979, 1981), which is considered to be in superior agreement.

CHEMICAL COMPOSITION

The mineral was concentrated by magnetic, organic heavy liquid, and hand-picking methods. The purity of the final concentrate was estimated to be $>99\%$ on the basis of optical evaluation at $40\times$ magnification; a modal analysis performed on grain-mount thin-sections under transmitted light gave a value of 99.1(5)%. The major impurity was ferro-actinolite, intimately replacing ferri-clinoholmquistite.

Wavelength-dispersive analyses were obtained using a CAMECA Camebax SX-50 electron microprobe at the University of Oviedo. Standard operating conditions were excitation voltage of 15 Kv, specimen current of 15 nA, peak-count time of 10 s, and background-count time of 5 s. The following standards and crystals were used for $K\alpha$ X-ray lines: albite for Si, Al, and Na; TAP for Si, Al, Mg, Na, and F; synthetic MnTiO₃ for Ti and Mn; andradite for Ca; PET for Ca, K, Cl and Ti; orthoclase for K; synthetic SrF₂ for F; vanadinite for Cl; sphalerite for Zn magnetite for Fe; synthetic MgO for Mg; and LiF for Fe, Mn, and Zn. Raw data were corrected for atomic number, absorption and fluorescence effects using the CAMECA (PAP) software. These analyses were complemented by

TABLE 3. Crystal structure refinement data

Space group	<i>c2/m</i>
<i>a</i> (Å)	9.480(4)
<i>b</i> (Å)	17.845(8)
<i>c</i> (Å)	5.283(7)
β (°)	102.03(9)
<i>V</i> (Å ³)	874.1(1)
Collected ref.	1487
Unique ref.	1320
Observed $I \geq 3\sigma$	1030
R_m (%)	2.5
R_F (%)	2.7
RW_F (%)	2.8
Crystal dimensions	$0.3 \times 0.1 \times 0.08$ mm
Diffractometer	Enraf-Nonius CAD4
Radiation	MoK α
Scan technique	$\theta/2\theta$
Scanwidth (°)	1.3
θ range (°)	1–30
Decay	$\leq 2\%$ variation
Abs. correct	$0.87 \div 1.18$

Li determination on the mineral concentrate by flame photometry.

The formula unit was calculated assuming the tetrahedral sites to be fully occupied by Si, as suggested by the structure refinement. The H₂O content was calculated on the basis of OH + F = 2 pfu. These assumptions allow calculations of Fe³⁺/Fe²⁺ ratios. A redox volumetry was carried out, yielding an FeO content of 10.8 ± 0.6 wt%, which agrees with the calculated result of 11.29 (57) wt%, confirming the validity of our assumptions. The average of six representative analyses, as well as the formula unit, are given in Table 1. The estimated standard deviations gives a measure of the intergranular variability in the sample.

CRYSTALLOGRAPHY

Experimental techniques

The powder diffraction pattern for the bulk sample was recorded on a Phillips PW1840 diffractometer. The ex-

perimental conditions were CuK α radiation, receiving slit of 0.1 mm, step size of 0.010 (°2 θ), and scan speed of 0.005 (°2 θ /s). NBS Si was used as a standard. Peak position calculations were carried out with the Phillips PC-APD software. Unit-cell parameters were refined from the corrected *d* values using the program UNITCELL (Holland and Redfern 1997). The indexed powder-pattern and refined unit-cell parameters are given in Table 2.

One crystal was selected for crystal-structure refinement on the basis of the morphology, absence of solid micro-inclusions and ferro-actinolite replacement. The aspects of the experimental procedure are given in Table 3. Unit-cell parameters were refined by least-squares, fitting the θ values of 25 reflections collected at 2θ angles from 13 to 42°. Intensities of the collected reflections were corrected for Lorentz and polarization effects. The centrosymmetric space group *C2/m* was confirmed during the course of the structure solution. Refinement was done without chemical constraints. Neutral atom scattering factors were used. These and anomalous dispersion corrections for heavy atoms were taken from the *International Tables for X-ray Crystallography* (Ibers and Hamilton 1974). All the structural sites were considered fully occupied except for the A site. The choice of the scattering factors, on the basis of chemical data and first refinement data, were Na and vacancy for A; Li and Na for M4; Si for T1 and T2; Fe and Mg for M1; Fe for M2; and Li and Fe for M3. An empirical absorption correction (Walker and Stuart 1983) was applied at the end of the isotropic refinements. Full-matrix least-squares refinement minimizing R_w ($[\sum w(|F_{obs}| - |F_{calc}|)^2 / \sum |F_{obs}|^2]^{1/2}$) with unit weights led to a value of 0.028. Only reflections with $I \geq 3\sigma$, were considered as observed. All the atomic positions but M4', Am, and H were refined using anisotropic thermal factors. Final difference-Fourier synthesis did not show electron density higher than 0.51 eÅ⁻³.

TABLE 4. Final parameters for the crystal-structure refinement

	<i>X/a</i>	<i>Y/b</i>	<i>Z/c</i>	m.a.n	U_{eq}	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
T1	0.2858(1)	0.0863(0)	0.2779(1)	14.0	0.56(4)	6.0(3)	5.7(3)	5.9(3)	-0.1(2)	1.0(3)	0.1(2)
T2	0.2968(1)	0.1712(0)	0.7870(1)	14.0	0.57(4)	6.3(3)	6.4(4)	6.0(3)	1.0(2)	1.2(3)	-0.1(1)
M1	0.0000(0)	0.0881(0)	0.5000(0)	16.1	0.86(7)	9.8(4)	9.0(4)	9.3(4)	0(0)	3.2(3)	0(0)
M2	0.0000(0)	0.1812(0)	0.0000(0)	24.7	0.60(4)	5.8(3)	5.9(3)	6.5(3)	0(0)	1.5(2)	0(0)
M3	0.0000(0)	0.0000(0)	0.0000(0)	13.5	0.86(9)	11(1)	6(1)	9(1)	0(0)	1.4(6)	0(0)
M4	0.0000(0)	0.2721(10)	0.5000(0)	4.1	2.2(4)	19(6)	30(8)	18(5)	0(0)	5(4)	0(0)
M4'	0.0000(0)	0.2489(9)	0.5000(0)		1.6(4)						
Am	0.0787(11)	0.5000(0)	0.1566(19)	2.8	1.0(2)						
Am'	0.0336(33)	0.5000(0)	0.0739(56)		1.9(6)						
O1	0.1119(2)	0.0910(1)	0.2045(4)		0.9(1)	8(1)	8(1)	11(1)	0(1)	1(1)	0(1)
O2	0.1214(2)	0.1735(1)	0.7316(4)		0.9(1)	8(1)	10(1)	9(1)	1(1)	1(1)	0(1)
O3	0.1170(5)	0.0000(0)	0.7037(9)		0.81(9)	5(2)	8(2)	10(1)	0(0)	-1(2)	0(0)
O4	0.3745(2)	0.2504(1)	0.7819(4)		1.2(9)	12(1)	10(1)	12(1)	-4(1)	0(1)	-1(1)
O5	0.3547(2)	0.1295(1)	0.0642(4)		1.1(9)	9(1)	15(1)	10(1)	-1(1)	3(1)	4(1)
O6	0.3464(2)	0.1203(1)	0.5631(4)		1.2(9)	11(1)	19(1)	8(1)	1(1)	2(1)	-5(1)
O7	0.3394(3)	0.0000(0)	0.2803(6)		1.1(9)	11(1)	7(1)	16(1)	0(0)	4(1)	0(0)
H	0.214(0)	0.0000(0)	0.746(0)		3.4(0)						

Notes: m.a.n. = mean atomic number, U_{eq} in Å² × 10², U_{ij} in Å² × 10³. Site-group population: T1 = 4Si; T2 = 4Si; M1 = 1.21 Mg ± 0.79 Fe²⁺; M2 = 1.54 Fe³⁺ + 0.20 Al + 0.12 Ti + 0.12 Fe²⁺ + 0.02 Zn; M3 = 0.49 Li + 0.44 Fe²⁺ + 0.07 Mn; M4 = 1.66 Li + 0.30 Na + 0.04 Ca; Am = 0.44 Na + 0.03K; O3 = 1.58 OH + 0.42 F.

TABLE 5. Selected interatomic distances

T1-O1	1.615	T2-O2	1.628
T1-O5	1.614	T2-O4	1.597
T1-O6	1.615	T2-O5	1.632
T1-O7	1.620	T2-O6	1.636
⟨T1-O⟩	1.616	⟨T2-O⟩	1.623
M1-O1 × 2	2.063	M3-O1 × 4	2.109
M1-O2 × 2	2.135	M3-O3 × 2	2.099
M1-O3 × 2	2.89	⟨M3-O⟩	2.106
⟨M1-O⟩	2.096	(M4)	(M4')
M2-O1 × 2	2.099	M4-O2 × 2	2.309 2.011
M2-O2 × 2	2.009	M4-O4 × 2	2.129 2.091
M2-O4 × 2	1.914	M4-O5 × 2	2.993 (3.253)
⟨M2-O⟩	2.007	M4-O6 × 2	2.474 2.807
		⟨M4-O⟩	2.476 2.507
Am-O5 × 2	2.722	Am'-O5 × 2	2.705
Am-O5 × 2	3.107	Am'-O5 × 2	2.861
Am-O6 × 2	2.621	Am'-O6 × 2	2.945
Am-O7	2.914	Am'-O7	3.373
Am-O7	2.584	Am'-O7	2.427
Am-O7	2.488	Am'-O7	2.327
⟨Am-O⟩	2.765	⟨Am'-O⟩	2.794

Note: Average e.s.d. for distances is 0.002 Å, except for the M4 and M4' positions, each of which are 0.009 Å.

Structure and site population

Refined structural data are given in Tables 4 and 5. Observed and calculated structure-factors are given in Table 6¹. This information, together with chemical data, allows us to outline the site populations.

The short mean bond lengths (m.b.l.) of the T1 and T2 tetrahedra (1.616 and 1.618 Å, respectively, Table 5) imply complete Si⁴⁺ occupancy in both sites on the basis of experimental data of C2/m amphiboles (Hawthorne 1983; Oberti et al. 1995a). This information has been used in calculating the formula unit from the chemical data.

Concerning the C-group cations, the observed m.b.l. (2.009 Å, Table 5) and mean atomic number (m.a.n.) (24.7, Table 4), when compared to the chemical data, imply complete ordering of high-charged cations and Zn at the M2 site (Hawthorne et al. 1993). There is general agreement concerning Al ordering at M2 in ^[4]Al-poor amphiboles (Oberti et al. 1995b), whereas Ti is known to enter the M1 or M2 sites in amphiboles (Hawthorne et al. 1994). However the presence of Ti at M1 is related to dehydrogenation, which is not the case of this amphibole (Oberti et al. 1992).

At the M1 site, the observed m.b.l. and m.a.n. are in agreement with Fe and Mg occupancy as calculated using the equations of Oberti et al. (1993), assuming an F content of 0.42 apfu, and bond lengths supplied by Hawthorne et al. (1993). At M3 site, the longer m.b.l. and the low m.a.n. (13.5) suggest complete ordering of ^[6]Li at this site, together with Fe²⁺ and minor Mn. The M4 site is split, being displaced the second position (called M4') approximately 0.41 Å along the twofold axis toward the

octahedral strip. The observed coordination allows us to assign the smaller cation, Li, to M4' and the larger, Na, to M4 (Oberti et al. 1995b). The A site has been refined using the split A-site model (Hawthorne 1983). Two maxima have been located, both of them in the Am site, from the difference-Fourier synthesis. A final difference-Fourier map with $\sin\theta/\lambda < 0.5$ in the O3 area shows a maximum at 0.9 Å from this site. This was assigned to H.

The proposed site-occupancy, on the basis of the above considerations and on the chemical data, is given in Table 4. Some differences occur between the observed (95.1) and calculated (98.7) m.a.n. at the octahedral strip. Two probable reasons exist for this disagreement. First, the crystal quality limits accuracy in structure refinement. Second, inter-granular chemical variability precludes a strict comparison because of the impossibility of analyzing the refined crystal. Minor chemical variations in the analyzed crystal, basically slightly more Mg than given in the formula unit, account for the main observed differences, especially at the M1 site.

RELATIONSHIPS TO OTHER AMPHIBOLE COMPOSITIONS

Ideal end-member ferri-clinoholmquistite has the formula $\text{Li}_2(\text{Fe}^{2+}, \text{Mg})_3\text{Fe}_2^{3+}\text{Si}_8\text{O}_{22}(\text{OH})_2$. This is related to end-member clinoholmquistite by the homovalent substitution $\text{Fe}^{3+} \leftrightarrow \text{Al}$. Moreover, although clinoholmquistites are presently classified in the Fe-Mg-Mn amphibole group, some of their crystal-chemical features suggest important analogies with alkali amphiboles, namely ^(B)Na-amphiboles.

Foremost, scattered and partial chemical data (Caballero 1993) point to extensive Na as a B-group cation (to ~0.7 apfu), recording the existence of, at least partial, solid solution between ferri-clinoholmquistite and other ^BNa-amphiboles (riebeckite and leakeite types). In addition, the investigated amphibole is also characterized by a significant presence of ^[6]Li at the M3 site (0.49 apfu). This feature has been often observed in alkali amphiboles (Borley 1963; Addinson and White 1968; Whittaker 1971; Lyons 1976; Hawthorne et al. 1992, 1996); in particular, it should be always associated with monovalent cations at M4, significant A-site occupancy, no ^[4]Al and high Fe³⁺ contents because of bond-valence and geometrical requirements (Hawthorne et al. 1993; Hawthorne et al. 1994). This is also the case for ferri-clinoholmquistite.

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- ¹ For a copy of Table 6, Document AM-98-002, contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. Deposit items may also be available on the American Mineralogist web site (see inside back cover of a current for web address).
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