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Ferro-ferri-hornblende from the Traversella mine (Ivrea, Italy): occurrence, mineral description and crystal-chemistry

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ABSTRACT

Ferro-ferri-hornblende is a new member of the amphibole supergroup (IMA-CNMNC 2015–054). It has been found in a rock specimen from the historical collection of Leandro De Magistris, which was collected at the Traversella mine (Val Chiusella, Ivrea, Piemonte, Italy). The specimen was catalogued as 'speziaite', and contains a wide range of amphibole compositions from tremolite/actinolite to magnesio-hastingsite. The end-member formula of ferro-ferri-hornblende is $^{A}\square^{B}Ca_2{}^{C}(Fe^{2+}_4Fe^{3+})^{T}(Si_7Al)\ O_{22}{}^{W}(OH)_2$, which requires SiO_2 43.41, Al_2O_3 5.26, FeO 29.66, Fe_2O_3 8.24 CaO 11.57, H_2O 1.86, total 100.00 wt.%. The empirical formula derived from electron microprobe analysis and single-crystal structure refinement for the holotype crystal is $^{A}(Na_{0.10}K_{0.13})_{\Sigma=0.23}\ {}^{B}(Ca_{1.93}Na_{0.07})_{\Sigma=2.00}\ {}^{C}(Mg_{1.16}Fe^{2+}_{3.21}Mn_{0.06}Fe^{3+}_{0.45}Al_{0.12}Ti_{0.01})_{\Sigma=5.01}$ $^{T}(Si_{7.26}Al_{0.74})_{\Sigma=8.00}\ O_{22}\ {}^{W}(OH_{1.89}F_{0.01}Cl_{0.10})_{\Sigma=2.00}$. Ferro-ferri-hornblende is biaxial (–), with $\alpha = 1.697(2)$, $\beta = 1.722(5)$, $\gamma = 1.726(5)$ and 2V (meas.) = 35.7(1.4)°, 2V (calc.) = 43.1°. The unit-cell parameters are a = 9.9307(5), b = 18.2232(10), c = 5.3190(3) Å, β = 104.857(1)°, V = 930.40 (9) Å^3, Z = 2, space group $C2/m$. The a:b:c ratio is 0.545:1:0.292. The strongest eight reflections in the powder X-ray pattern [d values (in Å), I, (hkl)] are: 8.493, 100, (110); 2.728, 69, (151); 3.151, 47, (310); 2.555, 37, ($\bar{2}$02); 2.615, 32, (061); 2.359, 28, ($\bar{3}$51); 3.406, 26, (131); 2.180, 25, (261). Type material is deposited in the collections of the Museo di Mineralogia, Dipartimento di Scienze della Terra e dell'Ambiente, Università di Pavia, under the catalogue number 2015–01. Sample M/U15285 from the historical collection of Luigi Colomba, presently at the Museo Regionale di Scienze Naturali di Torino, was also checked, and the presence of ferro-ferri-hornblende was confirmed.

Keywords: ferro-ferri-hornblende, electron-microprobe analysis, crystal-structure refinement, Traversella mine, Italy.

Introduction

This paper describes a further achievement obtained during a systematic search to provide the mineral description of common members of the amphibole supergroup which still miss an official recognition by IMA-CNMNC. This project started after approval of the new scheme for amphibole classification and nomenclature (Hawthorne *et al*., 2012), which is connected strongly with amphibole crystal-chemistry, and will provide formal approval for amphibole species that are widespread in common rocks.

The name 'hornblende' was proposed in 1789 by Abraham Gottlieb Werner, who combined an old

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German term for dark minerals of no ore value with the term ‘blende’, meaning ‘to deceive’. This name has long been used as a group name for dark green to black amphiboles, mostly ferro-hornblende or magnesio-hornblende according to the nomenclature in force. Indeed, in the book ‘*Rock-Forming Minerals, Volume 2b, Double-chain Silicates*’ by Deer *et al.* (1997), the term ‘hornblende’ is used as a group name for all aluminous amphiboles in the calcium amphibole subgroup. In their report on amphibole nomenclature, Hawthorne *et al.* (2012) give the name ‘magnesio-hornblende’ to the amphibole composition $^{A}\square^{B}Ca_2{}^{C}(Mg_4Al){}^{T}(Si_7Al)\,O_{22}{}^{W}(OH)_2$.

Despite the frequent occurrence in Nature of these compositions, according to the latest version of the IMA list of minerals (September 2015), only two entries contain the root-name hornblende, and are the two grandfathered end-members magnesio-hornblende and ferro-hornblende. In February 2015, IMA-CNMNC approved the sole hornblende species with a complete mineral description, i.e. magnesio-ferro-fluoro-hornblende 2014–091 from Portoscuso (Sardinia; Oberti *et al*., 2016).

Mineral data for ferro-ferri-hornblende

Sample description

The holotype specimen described in this work comes from the skarns at the Traversella mine, val Chiusella, Ivrea, Piemonte, Italy. The mine has been exploited for iron ore since the XI century, but the first notes are from the Roman historian Titus Livius. During the second World War, the Traversella deposit was mined by the FIAT company. It was closed in 1971, and now can only be visited by tourists on guided tours. Mineral collecting is strictly prohibited.

The sample (Fig. 1*a*) was collected in the 1960 s by Leandro De Magistris, former honorary curator of the Genova Mineralogical Museum, and was later acquired by Renato and Adriana Pagano. It consists of aggregates of ferro-ferri-hornblende crystals embedded in a matrix of fibrous to acicular tremolite with minor quartz and calcite (as determined by powder XRD analysis). A significant inter-crystalline variation in the hastingsite component is observed, with some crystals falling in the compositional field of hastingsite and even of magnesio-hastingsite. The sample was catalogued as ‘speziate’, a mineral first described in 1914 by Luigi Colomba (1866–1944), Mineralogy professor at the Universities of Sassari, Genova and then Torino, and named ‘speziate’ to

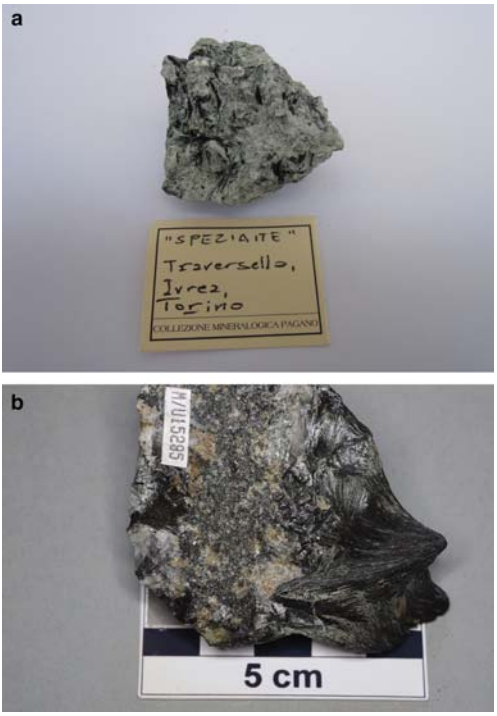

FIG. 1. (*a*) A picture of the holotype specimen studied in this work. The label is 3.7 cm. wide. (*b*) A picture of the old ‘speziaite’ sample M/U15285 described by Luigi Colomba in 1912.

honour Giorgio Spezia (1842–1911), also Mineralogy professor at the University of Torino, who in 1905 was the first to develop a method for the hydrothermal synthesis of quartz.

Colomba described ‘speziaite’ as aggregates of fibrous or acicular crystals, dark green or blackish in colour, occurring either in geodes or in druses at the Traversella mine. In the latter case, which seems to be the case for the specimen in this work, ‘speziaite’ is embedded in a fibrous whitish to greenish amphibole. The name ‘speziaite’, however, has never been approved by IMA; indeed, it was discredited (under its incorrect spelling ‘speziatite’) and redefined as hornblende by Leake (1978). After the official approval of the new species, we were able to examine the original sample from the Traversella mine used by Colomba to define ‘speziaite’, which is presently deposited in the mineralogical collection of the Museo Regionale di Scienze Naturali di Torino, Sezione di Mineralogia, Petrografia e Geologia (Torino) under the catalogue number M/U15285 (Fig. 1*b*), and found a very similar amphibole composition. Hence, former 'speziaite' is replaced definitively by ferro-ferri-hornblende.

The holotype (refined and analysed) crystal described in this work has the code 1260 in the amphibole database of the CNR-IGG Pavia. Type material is deposited in the collections of the Museo di Mineralogia, Dipartimento di Scienze della Terra e dell'Ambiente, Università di Pavia, under the catalogue number 2015–01. The sample in the mineralogical collection of the Museo Regionale di Scienze Naturali di Torino (refined crystal and two pieces of the same sample) should henceforth be considered a cotype.

In this paper, we also report on the chemical and structural data obtained for another crystal from the specimen belonging to the Pagano's collection, which is still ferro-ferri-hornblende but has a composition enriched in $^{A}R^{+}$ and $^{T,C}R^{3+}$ cations relative to that of the holotype crystal, i.e. it occurs in the part of the ferro-ferri-hornblende compositional space closer to hastingsite. This crystal has the code 1258 in the amphibole database of the CNR-IGG Pavia. This comparison is useful to describe crystal-chemical variation in the rock-specimen and to monitor their effects on polyhedron geometries.

Physical and optical properties

Ferro-ferri-hornblende occurs as acicular or lamellar crystals, is dark greenish, has vitreous lustre, is transparent and fluorescence is not present. The tenacity is brittle and single crystals show perfect cleavage parallel to {110}. The calculated density is 3.35 g/cm^3. Colomba (1914) measured the density of amphiboles in sample M/U15285 using methylene iodide at 12°C, and obtained 3.362 g/cm^3. A spindle stage was used to orient a crystal for measurement of refractive indices and 2V by extinction curves (Bartelmehs *et al*., 1992). The optical orientation was determined by transferring the crystal from the spindle stage to a single-crystal diffractometer and measuring the relative axial relations by X-ray diffraction (XRD). In transmitted light, ferro-ferri-hornblende is pleochroic, X = medium gold/brown (weakest), Y = dark brown/black (strongest), Z = dark grey (medium); $X \wedge a$ = 26.2° (β obtuse), $Y \parallel b$, $Z \wedge c$ = 11.5° (β acute). It is biaxial negative with indices of refraction $\alpha = 1.697(2)$, $\beta = 1.722(5)$, $\gamma = 1.726(5)$ measured with gel-filtered Na light (λ = 590 nm). 2V (meas.) = 35.7(4)°, 2V (calc.) = 43.1°.

Crystallography

Holotype ferro-ferri-hornblende from Traversella (1260) is monoclinic, space group $C2/m$, and has a = 9.9307(5), b = 18.2232(10), c = 5.3190(3) Å, β = 104. 857(1)º, V = 930.40(9) Å^3 (Z = 2). The a:b:c ratio calculated from the unit-cell parameters is 0.545:1:0.292. Diffraction data were collected for crystals 1260 and 1258 in the θ range 2–35° with a Bruker-AXS CCD diffractometer, working with graphite monochromatized Mo$K\alpha$ X-radiation (λ =

TABLE 1. Crystallographic details.

	Holotype ferro-ferri-hornblende 1260	Ferro-ferri-hornblende 1258	Cotype ferro-ferri-hornblende M/U15285 n.4
Size (μm)	210 × 100 × 60	200 × 80 × 60	392 × 144 × 63
a (Å)	9.9307(5)	9.9412(5)	9.9386(6)
b (Å)	18.2232(10)	18.2218(10)	18.2207(12)
c (Å)	5.3190(3)	5.3318(3)	5.3177(3)
β (°)	104.857(1)	104.946(1)	104.874(7)
V (Å^3)	930.40(9)	933.16(9)	930.70(10)
$a:b:c$	0.545:1:0.292	0.546:1:0.293	0.545:1:292
θ range (°)	2–35	2–35	2–36.6
R_{merge} × 100	1.6	1.6	2.8
R_{obs} × 100	2.5	2.9	3.5
R_{all} × 100	3.0	3.4	4.8
$\#_{collected}$	10,773	10,752	4355
Mean redund.	5	5	2
$\#_{all}$	2110	2120	2259

TABLE 2. Atomic coordinates, refined site-scattering values (ss, epfu), atom-displacement parameters (B_{eq}, Å^2;$\beta_{ij} \times 10^4$) for ferro-ferri-hornblende 1260, 1258 and M/U15285.

Site	ss	*x/a*	*y/b*	*z/c*	B_{eq}	β^{11}	β^{22}	β^{33}	β^{12}	β^{13}	β^{23}
1260											
O(1)		0.11015(12)	0.08865(6)	0.21398(21)	0.93(2)	24	8	81	−3	12	−1
O(2)		0.12079(9)	0.17450(3)	0.72604(33)	0.94(2)	23	8	85	−1	10	2
O(3)		0.11225(19)	0	0.71347(33)	1.06(3)	39	7	84	–	20	–
O(4)		0.36742(13)	0.24700(7)	0.79182(23)	1.11(2)	36	7	104	−4	22	−2
O(5)		0.34602(12)	0.13469(7)	0.09832(23)	1.13(2)	27	11	91	1	13	9
O(6)		0.34243(12)	0.11921(7)	0.59039(23)	1.07(2)	28	9	97	0	14	−6
O(7)		0.33333(19)	0	0.29115(36)	1.26(4)	34	6	156	–	18	–
T(1)		0.27963(4)	0.08455(2)	0.29672(8)	0.67(1)	20	5	60	−1	9	0
T(2)		0.28950(4)	0.17120(2)	0.80539(8)	0.72(1)	21	6	63	−1	12	0
M(1)	45.02(8)	0	0.08961(2)	$^1/_2$	0.78(1)	25	6	65	–	14	–
M(2)	43.63(8)	0	0.17880(2)	0	0.73(1)	22	5	72	–	13	–
M(3)	22.64(4)	0	0	0	0.77(1)	25	5	69	–	10	–
M(4)	38.88(54)	0	0.27907(13)	$^1/_2$	1.07(2)	34	7	122	–	35	–
A	0.99(3)	0	$^1/_2$	0	0.7(2)						
A(*m*)	1.21(7)	0.0271(28)	$^1/_2$	0.0679(57)	1.9(3)						
A(2)	0.75(7)	0	0.4682(17)	0	1.1(4)						
H	1.8	0.184(4)	0	0.749(8)	1.0						
M(4′)	0.93(4)	0	0.2594(42)	$^1/_2$	1.0						
1258											
O(1)		0.10779(14)	0.09004(8)	0.21379(25)	1.00(3)	25	9	86	−3	13	−1
O(2)		0.12103(14)	0.17577(8)	0.73027(26)	1.01(3)	24	8	91	0	9	3
O(3)		0.11438(23)	0	0.71372(38)	1.29(4)	64	6	77	–	30	–
O(4)		0.36792(15)	0.24799(8)	0.79179(27)	1.15(3)	37	8	108	−4	24	−2
O(5)		0.34694(14)	0.13652(8)	0.10171(27)	1.18(3)	29	11	95	0	10	10
O(6)		0.34230(14)	0.11956(8)	0.59638(27)	1.16(3)	28	9	114	1	15	−8
O(7)		0.33310(22)	0	0.29010(43)	1.41(4)	35	8	167	–	15	–
T(1)		0.27912(5)	0.08522(3)	0.29925(10)	0.75(1)	22	5	69	−1	9	0
T(2)		0.29018(5)	0.17216(3)	0.80880(9)	0.74(1)	20	5	68	−1	12	0
M(1)	46.56(11)	0	0.09038(2)	$^1/_2$	0.84(1)	27	6	68	–	15	–
M(2)	44.14(11)	0	0.17884(2)	0	0.75(1)	22	5	74	–	12	–
M(3)	24.00(5)	0	0	0	0.85(1)	27	5	76	–	10	–
M(4)	39.26(90)	0	0.28010(20)	$^1/_2$	1.09(3)	34	7	122	–	34	–
A	2.87(4)	0	$^1/_2$	0	1.1(1)						
A(*m*)	2.19(9)	0.0300(16)	$^1/_2$	0.0722(33)	1.6(2)						
A(2)	1.83(9)	0	0.4682(13)	0	2.4(3)						

H	1.8	0.184(5)	0	0.767(8)	1.0						
M(4′)	0.63(8)	0	0.2638(69)	1/2	1.0						
M/U15285											
O(1)		0.11052(12)	0.08864(8)	0.2132(2)	0.79	19	8	61	−2	8	−2
O(2)		0.12117(12)	0.17439(8)	0.7252(2)	0.81	17	7	75	−1	4	3
O(3)		0.11150(19)	0	0.7127(3)	0.82	22	6	75	–	10	–
O(4)		0.36739(12)	0.24674(8)	0.7924(2)	0.94	30	6	94	−3	18	−3
O(5)		0.34546(11)	0.13430(9)	0.0968(2)	0.96	22	10	73	1	9	9
O(6)		0.34215(11)	0.11920(8)	0.5887(2)	0.88	22	8	76	0	8	−7
O(7)		0.33269(17)	0	0.2917(4)	1.02	29	5	130	–	14	–
T(1)		0.27980(5)	0.08433(3)	0.29558(9)	0.43	13	3	37	−1	5	0
T(2)		0.28981(4)	0.17083(3)	0.80469(9)	0.56	16	4	49	−1	9	0
M(1)	46.46(7)	0	0.08953(2)	1/2	0.60	20	4	46	–	10	–
M(2)	45.70(7)	0	0.17904(2)	0	0.61	19	4	57	–	9	–
M(3)	23.18(8)	0	0	0	0.60	20	4	50	–	6	–
M(4)	39.95(8)	0	0.27871(3)	1/2	0.98	30	6	109	–	32	–
A	1.39(18)	0.032(7)	1/2	0.076(16)	2.01						
A(*m*)	0.6(3)	0	1/2	0	0.16						
H	2	0.179(4)	0	0.748(6)	0.35						

0.7107 Å). Omega rotation frames (scan width 0.3°, scan time 20 s, sample-to-detector distance 50 mm) were processed with the *SAINT* software (Bruker, 2003) and intensities were corrected for Lorentz and polarization effects; absorption effects were empirically evaluated by the *SADABS* software (Sheldrick, 1996) and an absorption correction was applied to the data. Only the reflections with Io > $3\sigma_I$ were considered as observed during unweighted full-matrix least-squares refinement on *F*. Scattering curves for fully ionized chemical species were used at sites where chemical substitutions occur; neutral *vs.* ionized scattering curves were used at the *T* and anion sites [except O(3)]. The first residuals in the difference-Fourier map (with peaks corresponding to 3 e/Å^2 for sample 1258 and 1.5 e/Å^2 for crystal 1260) are placed close to O(3), and are related to the presence of 0.10 Cl atoms per formula unit (apfu) (cf. Oberti *et al.*, 1993 for more details).

Ferro-ferri-hornblende from sample M/U15285 is monoclinic, space group *C*2/*m*, and has a = 9.9386(6), b = 18.2207(12), c = 5.3177(3) Å, β = 104.874(1)°, V = 930.7(1) Å^3 (Z = 2). Diffraction data were collected in the θ range 4–36.6° at CrisDi (Torino) using an Oxford Gemini R Ultra diffractometer equipped with a CCD area detector, with graphite-monochromatized Mo*K*α radiation (λ = 0.7107 Å). Omega rotation frames (scan width 1°, scan time 22 s, sample-to-detector distance 53 mm) were processed with the *CrysAlisPro*, Agilent technologies (version 1.171.36.24) and intensities were corrected for Lorentz and polarization effects. Data were corrected for empirical absorption using spherical harmonics (*Abspack*, Agilent ®). All reflections with Io > $2\sigma_I$ were considered as observed during weighted full-matrix least-squares refinement on F^2. Scattering curves were chosen according to the calculated chemical formulae.

For all the samples examined, crystallographic details are given in Table 1. Atom coordinates and displacement parameters, refined site-scattering values (Hawthorne *et al.*, 1995), and selected bond lengths and angles are given in Tables 2 and 3.

Powder XRD data (CuKα, λ = 1.54178 Å) were obtained for the holotype crystal 1260 using the *XPREP* utility of *SAINT* (Bruker, 2003), which generates a 2D powder diffractogram (Debye-Scherrer technique) starting from the F_{obs} collected on the single-crystal and taking into account solely the information concerning the unit-cell dimensions and the Laue symmetry. No Lorentz and polarization correction was applied. Data are given in Table 4. Observed structure factors together with

TABLE 3. Selected interatomic distances (Å) and interatomic angles (°) in the double chain of tetrahedra in ferro-ferri-hornblende 1260, 1258 and M/U15285.

	1260	1258	M/U15285		1260	1258	M/U15285
T(1)–O(1)	1.629(1)	1.648(1)	1.628(1)	*T*(2)–O(2)	1.621(1)	1.626(1)	1.621(1)
T(1)–O(5)	1.655(1)	1.675(1)	1.651(1)	*T*(2)–O(4)	1.594(1)	1.597(1)	1.593(1)
T(1)–O(6)	1.652(1)	1.668(1)	1.650(1)	*T*(2)–O(5)	1.654(1)	1.651(1)	1.650(1)
T(1)–O(7)	1.633(1)	1.648(1)	1.626(1)	*T*(2)–O(6)	1.670(1)	1.665(1)	1.668(1)
<*T*(1)–O>	1.642	1.660	1.639	<*T*(2)–O>	1.635	1.635	1.633
M(1)–O(1) × 2	2.089(1)	2.079(1)	2.096(1)	*M*(2)–O(1) × 2	2.134(1)	2.106(1)	2.137(1)
M(1)–O(2) × 2	2.130(1)	2.148(1)	2.130(1)	*M*(2)–O(2) × 2	2.113(1)	2.100(1)	2.120(1)
M(1)–O(3) × 2	2.133(1)	2.153(1)	2.127(1)	*M*(2)–O(4) × 2	2.010(1)	1.996(1)	2.010(1)
<*M*(1)–O>	2.117	2.127	2.118	<*M*(2)–O>	2.086	2.067	2.089
M(3)–O(1) × 4	2.113(1)	2.124(1)	2.112(1)	*M*(4)–O(2) × 2	2.403(2)	2.410(3)	2.399(1)
M(3)–O(3) × 2	2.106(2)	2.126(2)	2.105(1)	*M*(4)–O(4) × 2	2.327(1)	2.337(2)	2.328(1)
<*M*(3)–O>	2.111	2.124	2.110	*M*(4)–O(5) × 2	2.774(2)	2.732(2)	2.789(1)
				M(4)–O(6) × 2	2.550(2)	2.546(3)	2.554(1)
A–O(5) × 4	3.008(1)	3.037(2)	3.003(2)	<*M*(4)–O>	2.514	2.506	2.518
A–O(6) × 4	3.188(1)	3.177(1)	3.194(2)				
A–O(7) × 2	2.540(2)	2.543(2)	2.549(2)	*M*(4′)–O(2) × 2	2.13(6)	2.18(9)	
<*A*–O>	2.987	2.994	2.989	*M*(4′)–O(4) × 2	2.28(1)	2.29(1)	
				M(4′)–O(5) × 2	2.99(5)	2.91(8)	
A(*m*)–O(5) × 2	3.07(2)	3.11(1)	3.09(4)	*M*(4′)–O(6) × 2	2.82(6)	2.77(9)	
A(*m*)–O(5) × 2	2.99(1)	3.02(1)	2.98(2)	< *M*(4′)–O>	2.56	2.54	
A(*m*)–O(6) × 2	2.91(2)	2.89(1)	2.89(6)				
A(*m*)–O(7)	2.51(2)	2.52(2)	2.53(4)	*A*(2)–O(5) × 2	2.56(2)	2.58(2)	
A(*m*)–O(7)	3.34(3)	3.32(2)	3.29(4)	*A*(2)–O(6) × 2	2.83(2)	2.81(1)	
A(*m*)–O(7)	2.63(2)	2.63(1)	2.64(5)	*A*(2)–O(7) × 2	2.61(1)	2.61(1)	
<*A*(*m*)–O>	2.94	2.95	2.93	<*A*(2)–O>	2.66	2.67	
T(1)–O(5)–*T*(2)	136.7(1)	136.0(1)	137.2(1)	O(5)–O(6)–O(5)	167.8(1)	166.6(1)	168.1(1)
T(1)–O(6)–*T*(2)	139.1(1)	139.0(1)	139.5(1)	O(6)–O(7)–O(6)	108.3(1)	107.0(1)	108.7(1)
T(1)–O(7)–*T*(1)	141.3(1)	140.9(1)	141.9(1)				

TABLE 4. Powder X-ray data for ferro-ferri-hornblende 1260.

I_{rel}	*d*(calc)	*h k l*	I_{rel}	*d*(calc)	*h k l*	I_{rel}	*d*(calc)	*h k l*	I_{rel}	*d*(calc)	*h k l*
9	9.110	0 2 0	8	2.831	3 3 0	6	2.199	1 7 1	7	1.699	$\bar{2}$ 8 2
100	**8.493**	**1 1 0**	17	2.757	$\bar{3}$ 3 1	**25**	**2.180**	**2 6 1**			$\bar{1}$ 3 3
11	4.920	$\bar{1}$ 1 1	**69**	**2.728**	**1 5 1**	14	2.057	2 0 2	16	1.663	4 6 1
11	4.556	0 4 0	**32**	**2.615**	**0 6 1**	23	2.033	$\bar{4}$ 0 2	5	1.652	4 8 0
7	3.910	$\bar{1}$ 3 1	**37**	**2.555**	**$\bar{2}$ 0 2**			3 5 1	11	1.633	1 11 0
26	**3.406**	**1 3 1**	5	2.402	3 5 0	5	1.895	$\bar{4}$ 6 1	5	1.599	6 0 0
14	3.304	2 4 0	**28**	**2.359**	**$\bar{3}$ 5 1**	4	1.882	$\bar{1}$ 9 1	15	1.590	$\bar{1}$ 5 3
47	**3.151**	**3 1 0**	7	2.319	$\bar{1}$ 7 1	4	1.763	$\bar{5}$ 1 2	4	1.565	4 0 2
13	2.961	2 2 1	16	2.296	$\bar{3}$ 1 2				7	1.548	$\bar{6}$ 0 2

The eight strongest lines are in bold.

the cif have been deposited with the Principal Editor of *Mineralogical Magazine* and are available from http://www.minersoc.org/pages/e_journals/dep_mat_mm.html.

Electron microprobe analyses

Chemical analyses on crystals 1260 and 1258 used for structure refinement were carried out with a Cameca SX-100 electron microprobe (wavelength-dispersive spectroscopy mode, 15 kV, 20 nA, counting time 20 s, 5 μm beam diameter). The standards used are: Si and Ca: diopside (TAP); Ti: titanite (LPET); Al: andalusite (TAP); Fe: fayalite (LLiF); Mn: spessartine (LLiF); Mg: forsterite (LTAP); Zn: gahnite (LLiF); Na: albite (TAP); K: orthoclase (LPET); F: fluoro-riebeckite (TAP); Cl: tugtupite (LPET). H_2O was estimated based on two (OH,F,Cl) apfu and taking into account the constraints from the structure refinement. The oxide wt.% and the calculated unit-formula are reported in Table 5. End-member ferro-ferri-hornblende has the formula $^{A}\square^{B}Ca_2{}^{C}(Fe^{2+}_4Fe^{3+})$ $^{T}(Si_7Al)O_{22}{}^{W}(OH)_2$, which requires SiO_2 43.41, Al_2O_3 5.26, FeO 29.66, Fe_2O_3 8.24, CaO 11.57, H_2O 1.86, total 100.00 wt.%.

The final $[1 - (K_P/K_C)]$ compatibility index for holotype ferro-ferri-hornblende 1260 is −0.029 (excellent).

Crystal chemistry

Site populations and chemical variability

The chemical analyses available for crystals 1260 and 1258 were combined with the refined site-scattering values (in electrons per formula unit, epfu) to obtain site populations (Hawthorne *et al*., 1995). They are reported in Table 6, together with a comparison between the observed mean bond-lengths (mbl) and those calculated from the site populations based on the values of the distinct <cat–O> optimized for amphiboles during extensive crystal chemical work at IGG-CNR-Pv (Mg: 2.078 Å, Al: 1.929 Å, Ti: 1.960 Å, Mn^{2+}: 2.173 Å, Fe^{2+}: 2.125 Å, Fe^{3+}: 2.025 Å). The agreement between the refined and calculated site-scattering values is excellent, and validates the averaged composition of the crystal, the recalculation of the unit formula, and the partitioning of cations among the different groups of sites.

Inspection of the geometrical variations reported in Tables 3 and 5 confirms the calculated amounts of ^{T}Al and its ordering at the *T*(1) site; the small increase in ^{T}Al in crystal 1258 decreases slightly, but significantly, the stretching (along *c*) of the double chain of tetrahedra, measured by the O(5)–O(6)–O(5) angle. As far as the C cations are concerned, the comparison of the observed and calculated distances reported in Table 5, in particular the shorter <*M*(2)–O> distance measured in crystal 1258, confirm the ordering of high-charged cations at the *M*(2) site, which is expected in ^{W}(OH,F,Cl) amphiboles (Hawthorne and Oberti, 2007, Oberti *et al*., 2007).

The chemical variability observed in the two crystals (which is representative of that observed in a total of eight crystals refined and analysed) indicates variation from tremolite/actinolite to magnesio-hastingsite/hastingsite, where an increasing amount of $^{T}R^{3+}$ is balanced by an increase in $^{C}R^{3+}$ and $^{A}R^{+}$ in nearly equal proportions. Indeed, hastingsitic compositions have been found in this rock specimen, and always occur as strongly zoned dark-green crystals. Tremolitic amphiboles have been also identified (based on powder XRD

TABLE 5. Chemical composition (10 points) and unit formula (based on 24 anions) for ferro-ferri-hornblende (1260 and 1258).

Oxide	Wt.%	Range	Oxide	Wt.%	Range		apfu		apfu
1260									
SiO_2	46.63(1.83)	44.59–49.09	H_2O**	1.82		Si	7.26	Na	0.07
TiO_2	0.05(2)	0.03–0.08	F	0.02(3)	0.00–0.10	Al	0.74	Ca	1.93
Al_2O_3	4.67(1.19)	2.88–5.74	Cl	0.38(3)	0.12–0.64	Sum T	8.00	Sum B	2.00
Fe_2O_3*	3.81		O = F,Cl	−0.09		Ti^{4+}	0.01	K	0.13
FeO*	24.65		Total	100.22		Al	0.12	Na	0.10
$[FeO]_{tot}$	[28.08(94)]	26.50–29.06				Fe^{3+}	0.45	Sum A	0.23
MnO	0.48(5)	0.40–0.58		Group site-scattering (epfu)		Mn^{2+}	0.06	OH^-	1.89
MgO	4.99(68)	4.30–6.03		obs (SREF)	calc (EMP)	Fe^{2+}	3.21	F^-	0.01
ZnO	0.03(3)	0.00–0.08	C	111.29	112.36	Mg	1.16	Cl^-	0.10
CaO	11.59(9)	11.40–11.73	B	39.81	39.37	Sum C	5.01	Sum W	2.000
Na_2O	0.56(15)	0.33–0.70	A	2.95	3.57				
K_2O	0.63(29)	0.34–0.96	Total	154.05	155.30				
1258									
SiO_2	42.87(1.55)	41.00–45.03	H_2O**	1.81		Si	6.72	Mn^{2+}	0.04
TiO_2	0.14(4)	0.11–0.21	F	0.02(2)	0.00–0.05	Al	1.28	Na	0.07
Al_2O_3	9.14(0.62)	8.56–10.17	Cl	0.38(3)	0.35–0.46	Sum T	8.00	Ca	1.89
Fe_2O_3*	3.70		O = F,Cl	−0.10		Ti^{4+}	0.02	Sum B	2.00
FeO*	25.21		Total	100.22		Al	0.40	K	0.25
$[FeO]_{tot}$	[28.54(32)]	27.94–28.99				Fe^{3+}	0.44	Na	0.23
MnO	0.40(3)	0.37–0.46		Group site-scattering (epfu)		Mn^{2+}	0.02	0.02	0.48
MgO	3.53(69)	2.55–4.38		obs (SREF)	calc (EMP)	Fe^{2+}	3.30	OH^-	1.89
ZnO	0.02(2)	0.00–0.05	C	114.70	113.22	Mg	0.82	F^-	0.01
CaO	11.26(34)	10	B	39.90	39.57	Sum C	5.00	Cl^-	0.10
Na_2O	1.07(9)	1.93–1.21	A	6.89	7.28			Sum W	2.000
K_2O	1.13(8)	1.05–1.27	Total	161.49	160.07				

*FeO:Fe_2O_3 ratio calculated from single-crystal structure-refinement results.
**Calculated based on 24 (O, OH, F, Cl) with (OH + F + Cl) = 2 apfu.

TABLE 6. Site populations in ferro-ferri-hornblende 1260 and 1258.

		site scattering (epfu)		bond distance (Å)	
Site	Site population (apfu)	refined	calculated	refined	calculated
1260					
T(1)	3.26 Si + 0.74 Al			1.642	1.641
T(2)	4 Si				
M(1)	0.48 Mg + 1.52 Fe^{2+}	45.02	45.28	2.117	2.114
M(2)	0.46 Mg + 0.97 Fe^{2+} + 0.12 Al + 0.45 Fe^{3+} + 0.01 Ti^{4+}	43.63	44.22	2.086	2.090
M(3)	0.22 Mg + 0.72 Fe^{2+} + 0.06 Mn	22.64	22.86	2.111	2.118
C cations		111.29	112.36		
B cations	1.93 Ca + 0.07 Na	39.81	39.37		
A cations	0.10 Na + 0.13 K	2.95	3.57		
W anions	1.89 OH + 0.10 Cl + 0.01 F				
1258					
T(1)	6.72 Si + 1.28 Al			1.660	1.657
T(2)	4 Si				
M(1)	0.42 Mg + 1.58 Fe^{2+}	46.56	46.12	2.127	2.115
M(2)	0.24 Mg + 0.90 Fe^{2+} + 0.40 Al + 0.44 Fe^{3+} + 0.02 Ti^{4+}	44.14	43.36	2.067	2.057
M(3)	0.16 Mg + 0.82 Fe^{2+} + 0.02 Mn	24.00	23.74	2.124	2.118
C cations		114.70	113.22		
B cations	1.89 Ca + 0.04 Mn^{2+} + 0.07 Na	39.90	39.57		
A cations	0.23 Na + 0.25 K	6.89	7.28		
W anions	1.89 OH + 0.10 Cl + 0.01 F				

analysis) in the white microcrystalline matrix embedding hornblende and hastingsite.

The results of the structure refinement of ferro-ferri-hornblende from sample M/U15285 (Table 5) show that it is very close in composition and in crystal-chemistry to crystal 1260. The absence of the *A*(2) and *M*(4′) subsites may be due to the different models used during the refinement. Indeed, the site-scattering values refined for M/U15258 are very similar to those of crystal 1260 and, together with refined mean bond distances, may indicate a composition only slightly richer in ^{C}Fe and poorer in ^{T}Al and ^{A}Na, and thus even slightly closer to the end-member composition [*M*(1): 46.47 *vs*. 45.02, *M*(2): 45.70 *vs*. 43.63, *M*(3): 23.18 *vs*. 22.64, total C: 115.35 *vs*. 111.29 epfu; total B: 39.95 *vs*. 39.81 epfu; total A: 1.99 *vs*. 2.95 epfu].

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