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

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(Article begins on next page)

ABSTRACT

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Ferro-ferri-hornblende is a new member of the amphibole supergroup (IMA-CNMNC 2015-054). It has been found in 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 ${}^A\Box {}^B\text{Ca}_2 {}^C(\text{Fe}^{2+}_4\text{Fe}^{3+}) {}^T(\text{Si}_7\text{Al}) \text{O}_{22} {}^W(\text{OH})_2$, 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 ${}^A(\text{Na}_{0.10}\text{K}_{0.13})_{\Sigma=0.23} {}^B(\text{Ca}_{1.93}\text{Na}_{0.07})_{\Sigma=2.00} {}^C(\text{Mg}_{1.16}\text{Fe}^{2+}_{3.21}\text{Mn}_{0.06}\text{Fe}^{3+}_{0.45}\text{Al}_{0.12}\text{Ti}_{0.01})_{\Sigma=5.01} {}^T(\text{Si}_{7.26}\text{Al}_{0.74})_{\Sigma=8.00} \text{O}_{22} {}^W(\text{OH}_{1.89}\text{F}_{0.01}\text{Cl}_{0.10})_{\Sigma=2.00}$. Ferro-ferri-hornblende is biaxial (-), with $\alpha = 1.697(2)$, $\beta = 1.722(5)$, $\gamma = 1.726(5)$ and $2V$ (meas.) = $35.7(1.4)^\circ$, $2V$ (calc.) = 43.1° . The unit-cell parameters are $a = 9.9307(5)$, $b = 18.2232(10)$, $c = 5.3190(3)$ Å, $\beta = 104.857(1)^\circ$, $V = 930.40$ (9) Å³, $Z = 2$, space group $C2/m$. The $a:b:c$ ratio is 0.545:1:0.292. The strongest eight reflections in the X-ray powder pattern [d values (in Å), I , (hkl)] are: 8.493, 100, (110); 2.728, 69, (151); 3.151, 47, (310); 2.555, 37, (-202); 2.615, 32, (061); 2.359, 28, (-351); 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.

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48 **Introduction**

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

56 The name "hornblende" was proposed in 1789 by Abraham Gottlieb Werner, who combined
57 an old German term for dark minerals of no ore value with the term "blende", meaning "to deceive".
58 This name has long been used as a group name for dark green to black amphiboles, mostly ferro-
59 hornblende or magnesio-hornblende according to the nomenclature in force. Indeed, in the book
60 "Rock-forming minerals, volume 2b, Double chain silicates" by Deer *et al.* (1997), the term
61 "hornblende" is used as a group name for all aluminous amphiboles in the calcium amphibole
62 subgroup. In their report on amphibole nomenclature, Hawthorne *et al.* (2012) give the name
63 "magnesio-hornblende" to the amphibole composition $A^{\square} B^{\square} Ca_2^C (Mg_4Al)^T (Si_7Al) O_{22} W(OH)_2$.

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

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MINERAL DATA FOR FERRO-FERRI-HORNBLLENDE

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Sample description

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

81 The sample was collected in the 1960s by Leandro De Magistris, former honorary curator of
82 the Genova Mineralogical Museum, and was later acquired by Renato and Adriana Pagano (Figure
83 1a). It consists of aggregates of ferro-ferri-hornblende crystals embedded in a matrix of fibrous to
84 acicular tremolite with minor quartz and calcite (as determined by XRPD analysis). A significant
85 inter-crystalline variation in the hastingsite component is observed, with some crystals falling in the
86 compositional field of hastingsite and even of magnesio-hastingsite. The sample was catalogued as
87 "speziatite", a mineral first described in 1914 by Luigi Colomba (1866-1944), Mineralogy professor
88 at the Universities of Sassari, Genova and then Torino, and named "speziatite" to honour Giorgio
89 Spezia (1842-1911), also Mineralogy professor at the University of Torino, who in 1905 was the
90 first to develop a method for the hydrothermal synthesis of quartz.

91 Colomba described "speziatite" as aggregates of fibrous or acicular crystals, dark green or
92 blackish in colour, occurring either in geodes or in druses at the Traversella mine. In the latter case,
93 which seems to be the case of the specimen of this work, "speziatite" is embedded in a fibrous
94 whitish to greenish amphibole. The name "speziatite", however, has never been approved by IMA;
95 indeed, it was discredited (under its incorrect spelling "speziatite") and redefined as hornblende by
96 Leake (1978). After the official approval of the new species, we were able to examine the original
97 sample from the Traversella mine used by Colomba to define "speziatite", which is presently
98 deposited in the mineralogical collection of the Museo Regionale di Scienze Naturali di Torino,

99 Sezione di Mineralogia, Petrografia e Geologia (Torino) under the catalogue number M/15285
100 (Figure 1b), and found a very similar amphibole composition. Hence, former "speziaite" is
101 definitely replaced by ferro-ferri-hornblende.

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

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

115

116 **Physical and optical properties**

117 Ferro-ferri-hornblende occurs as acicular or lamellar crystals, is dark greenish, has vitreous lustre, is
118 transparent, and fluorescence is not present. The tenacity is brittle and single crystals show perfect
119 cleavage parallel to $\{110\}$. The calculated density is 3.35 g/cm^3 . Colomba (1914) measured the
120 density of amphiboles in sample M/U15285 using methylene iodide at 12°C , and obtained 3.362
121 g/cm^3 . A spindle stage was used to orient a crystal for measurement of refractive indices and 2V by
122 extinction curves (Bartelmehs *et al.*, 1992). The optical orientation was determined by transferring
123 the crystal from the spindle stage to a single-crystal diffractometer and measuring the relative axial
124 relations by X-ray diffraction. In transmitted light, ferro-ferri-hornblende is pleochroic, $X = \text{medium}$

125 gold/brown (weakest), Y = dark brown/black (strongest), Z = dark grey (medium); $X \wedge a = 26.2^\circ$ (β
126 obtuse), $Y // b$, $Z \wedge c = 11.5^\circ$ (β acute). It is biaxial negative with indices of refraction $\alpha = 1.697(2)$,
127 $\beta = 1.722(5)$, $\gamma = 1.726(5)$ measured with gel-filtered Na light ($\lambda = 590$ nm). $2V$ (meas.) = $35.7(4)^\circ$,
128 $2V$ (calc.) = 43.1° .

129 **Crystallography**

130 Holotype ferro-ferri-hornblende from Traversella (1260) is monoclinic, space group $C2/m$,
131 and has $a = 9.9307(5)$, $b = 18.2232(10)$, $c = 5.3190(3)$ Å, $\beta = 104.857(1)^\circ$, $V = 930.40(9)$ Å³ ($Z =$
132 2). The $a:b:c$ ratio calculated from the unit cell parameters is 0.545:1:0.292. Diffraction data were
133 collected for crystals 1260 and 1258 in the θ range 2–35° with a Bruker-AXS CCD diffractometer,
134 working with graphite monochromatized MoK α X-radiation ($\lambda = 0.7107$ Å). Omega rotation frames
135 (scan width 0.3°, scan time 20 s, sample-to-detector distance 50 mm) were processed with the
136 SAINT software (Bruker, 2003) and intensities were corrected for Lorentz and polarization effects;
137 absorption effects were empirically evaluated by the SADABS software (Sheldrick, 1996) and an
138 absorption correction was applied to the data. Only the reflections with $I_o > 3\sigma_I$ were considered as
139 observed during unweighted full-matrix least-squares refinement on F . Scattering curves for fully
140 ionised chemical species were used at sites where chemical substitutions occur; neutral *vs* ionized
141 scattering curves were used at the T and anion sites [except O(3)]. The first residuals in the
142 Difference Fourier map (with peaks corresponding to 3 e/Å² for sample 1258 and 1.5 e/Å² for
143 crystal 1260) are placed close to O(3), and are related to the presence of 0.10 Cl apfu (cf. Oberti *et*
144 *al.* 1993 for more details).

145 Ferro-ferri-hornblende from sample M/U15285 is monoclinic, space group $C2/m$, and has a
146 = 9.9386(6), $b = 18.2207(12)$, $c = 5.3177(3)$ Å, $\beta = 104.874(1)^\circ$, $V = 930.7(1)$ Å³ ($Z = 2$).
147 Diffraction data was collected in the θ range 4–36.6° at CrisDi (Torino) using an Oxford Gemini R
148 Ultra diffractometer equipped with a CCD area detector, with graphite-monochromatized MoK α
149 radiation ($\lambda = 0.7107$ Å). Omega rotation frames (scan width 1°, scan time 22 s, sample-to-detector

150 distance 53 mm) were processed with the CrysAlis Pro, Agilent technologies (version 1.171.36.24)
151 and intensities were corrected for Lorentz and polarization effects. Data were corrected for
152 empirical absorption using spherical harmonics (Abspack, Agilent®). All reflections with $I_0 > 2\sigma_I$
153 were considered as observed during weighted full-matrix least-squares refinement on F^2 . Scattering
154 curves were chosen according to the calculated chemical formulae.

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

158 X-ray powder-diffraction data ($\text{CuK}\alpha$, $\lambda = 1.54178 \text{ \AA}$) were obtained for the holotype crystal
159 1260 using the XPREP utility of SAINT (Bruker, 2003), which generates a 2D powder
160 diffractogram (Debye-Scherrer technique) starting from the F_{obs} collected on the single-crystal and
161 taking into account solely the information concerning the unit-cell dimensions and the Laue
162 symmetry. No Lorentz and polarization correction was applied. Data are given in Table 4. Observed
163 structure factors have been deposited together with the cif files.

164 **EMP analyses**

165 Chemical analyses on crystals 1260 and 1258 used for structure refinement were done with a
166 Cameca SX-100 electron microprobe (WDS mode, 15 kV, 20 nA, counting time 20 s, 5 μm beam
167 diameter). The standards used are: Si and Ca: diopside (TAP); Ti: titanite (LPET); Al: andalusite
168 (TAP); Fe: fayalite (LLiF); Mn: spessartine (LLiF); Mg: forsterite (LTAP); Zn: gahnite (LLiF); Na:
169 albite (TAP); K: orthoclase (LPET); F: fluoro-riebeckite (TAP); Cl: tugtupite (LPET). H_2O was
170 estimated based on $2 = (\text{OH}, \text{F}, \text{Cl})$ apfu and taking into account the constraints from the structure
171 refinement. The oxide wt% and the calculated unit-formula are reported in Table 5. End-member
172 ferro-ferri-hornblende has the formula $^{\text{A}}\square ^{\text{B}}\text{Ca}_2 ^{\text{C}}(\text{Fe}^{2+}_4\text{Fe}^{3+}) ^{\text{T}}(\text{Si}_7\text{Al}) \text{O}_{22} ^{\text{W}}(\text{OH})_2$, which requires
173 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%.

174 The final $[1 - (K_P/K_C)]$ compatibility index for holotype ferro-ferri-hornblende 1260 is -
175 0.029 (excellent).

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Crystal chemistry

178 Site populations and chemical variability

179 The chemical analyses available for crystals 1260 and 1258 were combined with the refined site-
180 scattering values (in electrons per formula unit, epfu) to obtain site populations (Hawthorne *et al.*,
181 1995). They are reported in Table 6, together with a comparison between the observed mean bond-
182 lengths (mbl) and those calculated from the site populations based on the values of the distinct <cat-
183 O> optimized for amphiboles during extensive crystal chemical work at IGG-CNR-Pv (Mg: 2.078
184 Å, Al: 1.929 Å Ti: 1.960 Å, Mn²⁺: 2.173 Å, Fe²⁺: 2.125 Å, Fe³⁺: 2.025 Å). The agreement between
185 the refined and calculated site-scattering values is excellent, and validates the averaged composition
186 of the crystal, the recalculation of the unit formula, and the partitioning of cations among the
187 different groups of sites.

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

195 The chemical variability observed in the two crystals (which is representative of that
196 observed in a total of 8 crystals refined and analyzed) indicates variation from tremolite/actinolite to
197 magnesio-hastingsite/hastingsite, where an increasing amount of ^TR³⁺ is balanced by an increase in

198 ${}^{\text{C}}\text{R}^{3+}$ and ${}^{\text{A}}\text{R}^{+}$ in nearly equal proportions. Indeed, these latter compositions have been found in this
199 rock-specimen, and always occur as strongly zoned dark-green crystals. Tremolitic amphiboles
200 have been also identified (based on XRPD analysis) in the white microcrystalline matrix embedding
201 hornblende and hastingsite.

202 The results of the structure refinement of ferro-ferri-hornblende from sample M/U15285
203 (Table 5) show that it is very close in composition and in crystal-chemistry to crystal 1260. The
204 absence of the $A(2)$ and $M(4')$ subsites may be due to the different models used during the
205 refinement. Indeed, the site-scattering values refined for M/U15258 are very similar to those of
206 crystal 1260 and, together with refined mean bond distances, may indicate a composition only
207 slightly richer in ${}^{\text{C}}\text{Fe}$ and poorer in ${}^{\text{T}}\text{Al}$ and ${}^{\text{A}}\text{Na}$, and thus even slightly closer to the end-member
208 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
209 vs. 111.29 epfu; total B: 39.95 vs. 39.81 epfu; total A: 1.99 vs. 2.95 epfu].

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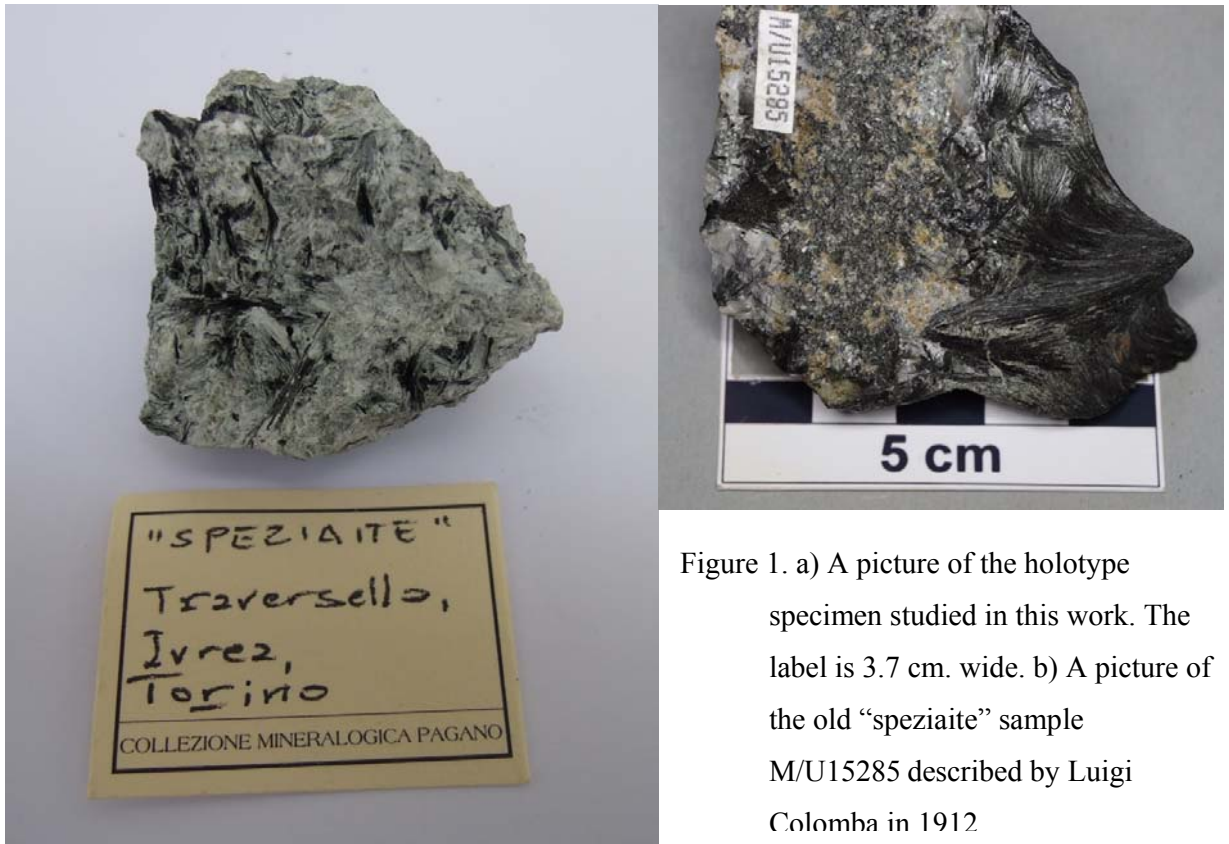


Figure 1. a) A picture of the holotype specimen studied in this work. The label is 3.7 cm. wide. b) A picture of the old “speziaite” sample M/U15285 described by Luigi Colomba in 1912

274 **TABLE 1.** Crystallographic details

	Holotype ferro-ferri- hornblende 1260	Ferro-ferri- hornblende 1258	Cotype ferro-ferri- hornblende M/U15285 n.4		Holotype ferro-ferri- hornblende 1260	Ferro-ferri- hornblende 1258	Cotype ferro-ferri- hornblende M/U15285 n.4
Size (μm)	210 x 100 x 60	200 x 80x 60	392 x 144 x 63	$R_{\text{merge}} \times 100$	1.6	1.6	2.8
a (\AA)	9.9307(5)	9.9412(5)	9.9386(6)	$R_{\text{obs}} \times 100$	2.5	2.9	3.5
b	18.2232(10)	18.2218(10)	18.2207(12)	$R_{\text{all}} \times 100$	3.0	3.4	4.8
c	5.3190(3)	5.3318(3)	5.3177(3)				
β ($^\circ$)	104.857(1)	104.946(1)	104.874(7)	$\#_{\text{collected}}$	10773	10752	4355
V (\AA^3)	930.40(9)	933.16(9)	930.70(10)	Mean redund.	5	5	2
$a:b:c$	0.545:1:0.292	0.546:1:0.293	0.545:1:292	$\#_{\text{all}}$	2110	2120	2259
θ range ($^\circ$)	2-35	2-35	2-36.6				

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276

277 **TABLE 2.** Atomic coordinates, refined site-scattering values (ss, epfu), atom-displacement parameters (B_{eq} ,
 278 $\text{\AA}^2; \beta_{ij} \times 10^4$) for ferro-ferri-hornblende 1260 and 1258.

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)	$\frac{1}{2}$	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)	$\frac{1}{2}$	0.98	30	6	109	-	32	-
A	1.39(18)	0.032(7)	$\frac{1}{2}$	0.076(16)	2.01						
A(m)	0.6(3)	0	$\frac{1}{2}$	0	0.16						
H	2	0.179(4)	0	0.748(6)	0.35						

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283**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
<i>T</i> (1)–O(1)	1.629(1)	1.648(1)	1.628(1)	<i>T</i> (2)–O(2)	1.621(1)	1.626(1)	1.621(1)
<i>T</i> (1)–O(5)	1.655(1)	1.675(1)	1.651(1)	<i>T</i> (2)–O(4)	1.594(1)	1.597(1)	1.593(1)
<i>T</i> (1)–O(6)	1.652(1)	1.668(1)	1.650(1)	<i>T</i> (2)–O(5)	1.654(1)	1.651(1)	1.650(1)
<i>T</i> (1)–O(7)	1.633(1)	1.648(1)	1.626(1)	<i>T</i> (2)–O(6)	1.670(1)	1.665(1)	1.668(1)
< <i>T</i> (1)–O>	1.642	1.660	1.639	< <i>T</i> (2)–O>	1.635	1.635	1.633
<i>M</i> (1)–O(1) ×2	2.089(1)	2.079(1)	2.096(1)	<i>M</i> (2)–O(1) ×2	2.134(1)	2.106(1)	2.137(1)
<i>M</i> (1)–O(2) ×2	2.130(1)	2.148(1)	2.130(1)	<i>M</i> (2)–O(2) ×2	2.113(1)	2.100(1)	2.120(1)
<i>M</i> (1)–O(3) ×2	2.133(1)	2.153(1)	2.127(1)	<i>M</i> (2)–O(4) ×2	2.010(1)	1.996(1)	2.010(1)
< <i>M</i> (1)–O>	2.117	2.127	2.118	< <i>M</i> (2)–O>	2.086	2.067	2.089
<i>M</i> (3)–O(1) ×4	2.113(1)	2.124(1)	2.112(1)	<i>M</i> (4)–O(2) ×2	2.403(2)	2.410(3)	2.399(1)
<i>M</i> (3)–O(3) ×2	2.106(2)	2.126(2)	2.105(1)	<i>M</i> (4)–O(4) ×2	2.327(1)	2.337(2)	2.328(1)
< <i>M</i> (3)–O>	2.111	2.124	2.110	<i>M</i> (4)–O(5) ×2	2.774(2)	2.732(2)	2.789(1)
<i>A</i> –O(5) ×4	3.008(1)	3.037(2)	3.003(2)	<i>M</i> (4)–O(6) ×2	2.550(2)	2.546(3)	2.554(1)
<i>A</i> –O(6) ×4	3.188(1)	3.177(1)	3.194(2)	< <i>M</i> (4)–O>	2.514	2.506	2.518
<i>A</i> –O(7) ×2	2.540(2)	2.543(2)	2.549(2)	<i>M</i> (4′)–O(2) ×2	2.13(6)	2.18(9)	
< <i>A</i> –O>	2.987	2.994	2.989	<i>M</i> (4′)–O(4) ×2	2.28(1)	2.29(1)	
<i>A</i> (<i>m</i>)–O(5) ×2	3.07(2)	3.11(1)	3.09(4)	<i>M</i> (4′)–O(5) ×2	2.99(5)	2.91(8)	
<i>A</i> (<i>m</i>)–O(5) ×2	2.99(1)	3.02(1)	2.98(2)	<i>M</i> (4′)–O(6) ×2	2.82(6)	2.77(9)	
<i>A</i> (<i>m</i>)–O(6) ×2	2.91(2)	2.89(1)	2.89(6)	< <i>M</i> (4′)–O>	2.56	2.54	
<i>A</i> (<i>m</i>)–O(7)	2.51(2)	2.52(2)	2.53(4)	<i>A</i> (2)–O(5) ×2	2.56(2)	2.58(2)	
<i>A</i> (<i>m</i>)–O(7)	3.34(3)	3.32(2)	3.29(4)	<i>A</i> (2)–O(6) ×2	2.83(2)	2.81(1)	
<i>A</i> (<i>m</i>)–O(7)	2.63(2)	2.63(1)	2.64(5)	<i>A</i> (2)–O(7) ×2	2.61(1)	2.61(1)	
< <i>A</i> (<i>m</i>)–O>	2.94	2.95	2.93	< <i>A</i> (2)–O>	2.66	2.67	
<i>T</i> (1)–O(5)– <i>T</i> (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)
<i>T</i> (1)–O(6)– <i>T</i> (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)
<i>T</i> (1)–O(7)– <i>T</i> (1)	141.3(1)	140.9(1)	141.9(1)				

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

<i>I</i> _{rel}	<i>d</i> (calc)	<i>h k l</i>	<i>I</i> _{rel}	<i>d</i> (calc)	<i>h k l</i>	<i>I</i> _{rel}	<i>d</i> (calc)	<i>h k l</i>	<i>I</i> _{rel}	<i>d</i> (calc)	<i>h k l</i>
9	9.110	0 2 0	8	2.831	3 3 0	6	2.199	1 7 1	7	1.699	-2 8 2
100	8.493	1 1 0	17	2.757	-3 3 1	25	2.180	2 6 1			-1 3 3
11	4.920	-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	-4 0 2	5	1.652	4 8 0
7	3.910	-1 3 1	37	2.555	-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	-4 6 1	5	1.599	6 0 0
14	3.304	2 4 0	28	2.359	-3 5 1	4	1.882	-1 9 1	15	1.590	-1 5 3
47	3.151	3 1 0	7	2.319	-1 7 1	4	1.763	-5 1 2	4	1.565	4 0 2
13	2.961	2 2 1	16	2.296	-3 1 2				7	1.548	-6 0 2

288 Note: The strongest eight lines are in bold.

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291 **TABLE 5.** Chemical composition (10 points) and unit formula (based on 24 anions) for ferro-ferri-hornblende
 292 (1260).

Oxide	wt%	Range	Oxide	wt%	Range	apfu		apfu	
1260									
SiO ₂	46.63(1.83)	44.59-49.09	H ₂ O**	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 ₂ O 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 ₂ O 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 ₂ O	0.56(15)	0.33-0.70	A	2.95	3.57				
K ₂ O	0.63(29)	0.34-0.96	Total	154.05	155.30				
1258									
SiO ₂	42.87(1.55)	41.00-45.03	H ₂ O**	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 ₂ O 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 ₂ O 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 ₂ O	1.07(9)	1.93-1.21	A	6.89	7.28			Sum W	2.000
K ₂ O	1.13(8)	1.05-1.27	Total	161.49	160.07				

293 * FeO:Fe₂O₃ ratio calculated from single-crystal structure-refinement results;

294 ** calculated based on 24 (O, OH, F, Cl) with (OH + F + Cl) = 2 apfu.

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298 **Table 6.** Site populations in ferro-ferri-hornblende 1260 and 1258.

Site	Site population (<i>apfu</i>)	site scattering (<i>epfu</i>)		bond distance (Å)	
		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				

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