

Tapiolite-(Fe): paragenesis and chemical compositions in pegmatites of the Pampean Pegmatite Province

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ABSTRACT

The paragenesis and chemical compositions of tapiolite-(Fe) present in different pegmatite occurrences from the Pampean Pegmatite Province are described. Exceptionally, the mineral occurs as an idiomorphic single crystal; more commonly, it is present associated with columbite-group minerals or, rarely, with wodginite-group minerals, where tapiolite-(Fe) usually shows textural evidence of replacement by these associated phases. The chemical composition shows wt.% ranges of Ta_2O_5 between 86.22 and 72.11, FeO from 14.03 to 10.46, and $Fe_2O_3_{calc.}$ from 2.06 to 0.00. The minor elements mean contents in wt.% are: Nb_2O_5 (2.40), TiO_2 (0.74), SnO_2 (0.21), and MnO (1.15). The trace elements have contents ≤ 0.5 wt.% or are negligible. The Ta# ($Ta / (Ta + Nb)$ at) varies from 0.99 to 0.84, with most of the samples clustering in tight groups. The Ta# values of tapiolite are higher than those of associated CGM and similar to those of WGM, while the Mn# values are always lower than in both mineral groups. The origin of the tapiolite-(Fe) in most of the studied samples is ascribed, as in other occurrences, to the late-stage magmatic crystallization in disequilibrium conditions of a fluxed and highly differentiated evolved pegmatite melt.

Keywords: chemical compositions, late-stage crystallization, Argentina.

RESUMEN

Tapiolita-(Fe): paragénesis y composición química en pegmatitas de la provincia pegmatítica Pampeana. Se describen la paragénesis y composición química de la mayoría de las manifestaciones de tapiolita-(Fe) en yacimientos de la Provincia Pegmatítica Pampeana. El mineral se encuentra excepcionalmente en cristales idiomorfos; más comúnmente se presenta asociado con minerales del grupo de la columbita o del grupo de la wodginita mostrando evidencias texturales de reemplazo por estas fases. Las composiciones químicas muestran rangos expresados en %p. de Ta_2O_5 comprendidos entre 86,22 y 72,11, FeO de 14,03 a 10,46, y de $Fe_2O_3_{calc.}$ desde 2,06 a 0,00. Los elementos menores tienen contenidos medios (%p.) de: Nb_2O_5 (2,40), TiO_2 (0,74), SnO_2 (0,21) y MnO (1,15). Los elementos traza tienen contenidos $\leq 0,5$ % p. o son ínfimos. Los valores de Ta# ($Ta / (Ta + Nb)$ at) de tapiolita-(Fe) varían de 0,99 a 0,84 y muestran muy buen agrupamiento en la mayoría de las pegmatitas. Siempre los valores de Ta# de tapiolita-(Fe) son más altos que en los análisis de los minerales del grupo de la columbita y similares a los del grupo de la wodginita asociados, mientras que los valores de Mn# son inferiores a los de ambos grupos. El origen de la tapiolita-(Fe), como en la mayoría de las diferentes paragénesis estudiadas, se adscribe a la cristalización magmática tardía y en desequilibrio de un fundido pegmatítico evolucionado, enriquecido en fundentes y elementos incompatibles.

Keywords: composiciones químicas, estadio de cristalización tardía, Argentina.

INTRODUCTION

The tetragonal (SG $P4_2/mnm$) oxides of Ta, Fe and Mn form the non-continuous series of the tapiolite group whose end members are tapiolite-(Fe) $FeTa_2O_6$ and tapiolite-(Mn) $MnTa_2O_6$. Tapiolite-(Fe) is relatively frequent in the most differentiated and high field strength elements (HFSE) enriched units of the rare-element pegmatites (Černý and Ercit 1989, Falster et al. 2011, Ryznar et al. 2023) and less frequent in highly evolved granites (e.g., Cheng et al. 1997, Chudík et al. 2008), where it is usually volumetrically subordinate to the columbite-group minerals (CGM) and wodginite-group minerals (WGM). Tapiolite-(Mn) is extremely rare.

In spite that tapiolite-(Fe) does not belong to the CGM because it has a trirutile structure (Chukanov et al. 2023), it is very common to represent the chemical composition of the associated phases in the columbite quadrilateral, where it covers approximately 5% of the quadrilateral surface. The crystal chemistry of tapiolite-(Fe) is a cation-ordered derivative of the rutile structure, but the degree of order is not a simple issue (Wise and Černý 1996, Zema et al. 2006, Ercit 2010). The relationships with the CGM minerals show a miscibility gap that was empirically and experimentally investigated (e.g., Turnock 1966, Černý et al. 1992, Van Lichtervelde et al. 2018).

In Argentina, tapiolite-(Fe) was mentioned in several occurrences situated in the Pampean Pegmatite Province, a major unit situated in northwestern and central Argentina (Galliski 1994a, b). However, the published analytical data is limited as well as the paragenetic relationships, therefore the aim of this paper is to contribute with the data obtained

during past investigations on this mineral at some of the main localities.

The information is particularly relevant, besides its mineralogical significance, because tapiolite-(Fe) has one of the highest grades in Ta_2O_5 , and tantalum is a critical element in the present circumstances.

LOCALITIES AND PARAGENESIS

The location and main features of the tapiolite-(Fe) occurrences are quoted in Table 1. Tapiolite-(Fe) was first

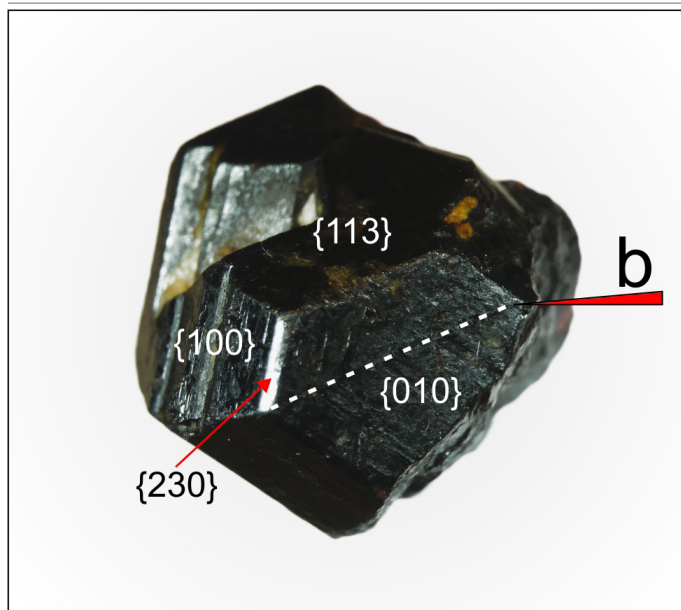


Figure 1. Euhedral crystal of tapiolite-(Fe) from Santa Elena pegmatite; the crystal size is 1.1 x 1.1 cm. The dashed line shows the b twin plane. The crystallographic notation is that of the crystallographic form to which the indicated face belongs.

Table 1. Localities and data of tapiolite-(Fe) in the Pampean pegmatite province

Occurrence	Latitude	Longitude	Paragenesis	O. M.	EDS	XRD	EPMA	LA-ICP-MS	References
Sta. Elena	-24.830136°	-66.342357°	5	x		x			2
			1,5,6	x			x		
El Peñón	-24.836309°	-66.329060°	1,6			x			2
Tres Tetas	-24.891175°	-66.314407°	1	x					2
Papachacra	~ -27.0000°	~ -67.0000°	5			x	x		9
Don Pancho	-32.180227°	-65.401199°	4,6			x			8
León Herido	-32.199939°	-65.352764°	1,4					x	6
Nancy	-32.480359°	-65.279777°	1,5				x	x	5
La Viquita	-32.856881°	-65.110948°	1, 2, 3	x		x			1.4
San Elías	-32.857918°	-65.118516°	1, 2, 3, 4, 5			x	x		3
Víctor Hugo	-33.077047°	-66.008394°	1, 6	x			x		10
Cañada Honda	-32.913611°	-65.988531°	1		x	x			7

Mineral symbols after IMA-CNMNC (Warr 2021). Paragenesis: 1 = CGM, 2 = Fwdg, 3 = Ftwdg, 4 = Ab, 5 = Qz, 6 = Mic. References: 1) Arcidiácono (1974), 2) Galliski (1983), 3) Galliski et al. (1999), 4) Galliski et al. (2008), 5) Galliski et al. (2019), 6) Galliski et al. (2021), 7) Rosello and Barbosa (1988), 8) Vistalli (1973), 9) Colombo et al. (2011), 10) This paper.

mentioned in the Pancho pegmatite (also known as Don Pancho), San Luis province, where it was identified by X-ray diffraction (XRD) (Vistalli 1973). In the same province, Arcidiácono (1974) identified this phase by optical and XRD means in La Viquita pegmatite. In El Quemado pegmatite district of Salta province, it was mentioned in the Santa Elena, El Peñón and Tres Tetras pegmatites (Galliski 1983). Rosello and Barbosa (1988) described tapiolite-(Fe) grains as part of the heavy mineral association of the Cañada Honda alluvial deposits, San Luis province. Later on it was mentioned in the San Elías and La Viquita pegmatites (Galliski et al. 1999, 2008), in Papachacra miarolitic pegmatites, Catamarca province (Colombo et al. 2011), and in Nancy and León Herido pegmatites, both in San Luis province (Galliski et al. 2019, 2021).

In El Quemado District of Salta province, tapiolite-(Fe) was found in a 1.1 x 1.1 cm euhedral crystal, possibly in the dumps of Santa Elena Central pegmatite, associated with a small piece of milky quartz (Fig. 1). In the same pegmatite, it occurs as an inclusion in a fragment of CGM that was also found in the dumps. In El Peñón and Tres Tetras pegmatites small grains of tapiolite-(Fe) occur included in centimetric grains of CGM in the inner part of the intermediate zones.

At Papachacra, tapiolite-(Fe) occurs in miarolitic cavities of an A-type granite in mm-sized crystals on microcline or albite. In the same locality and setting were mentioned: CGM, clinofergusonite-(Y), ilmenite, members of the pyrochlore supergroup (with both Ta- and Nb-dominant species), rutile, bastnäsite-(Ce), parisite-(Ce), siderite, florencite-

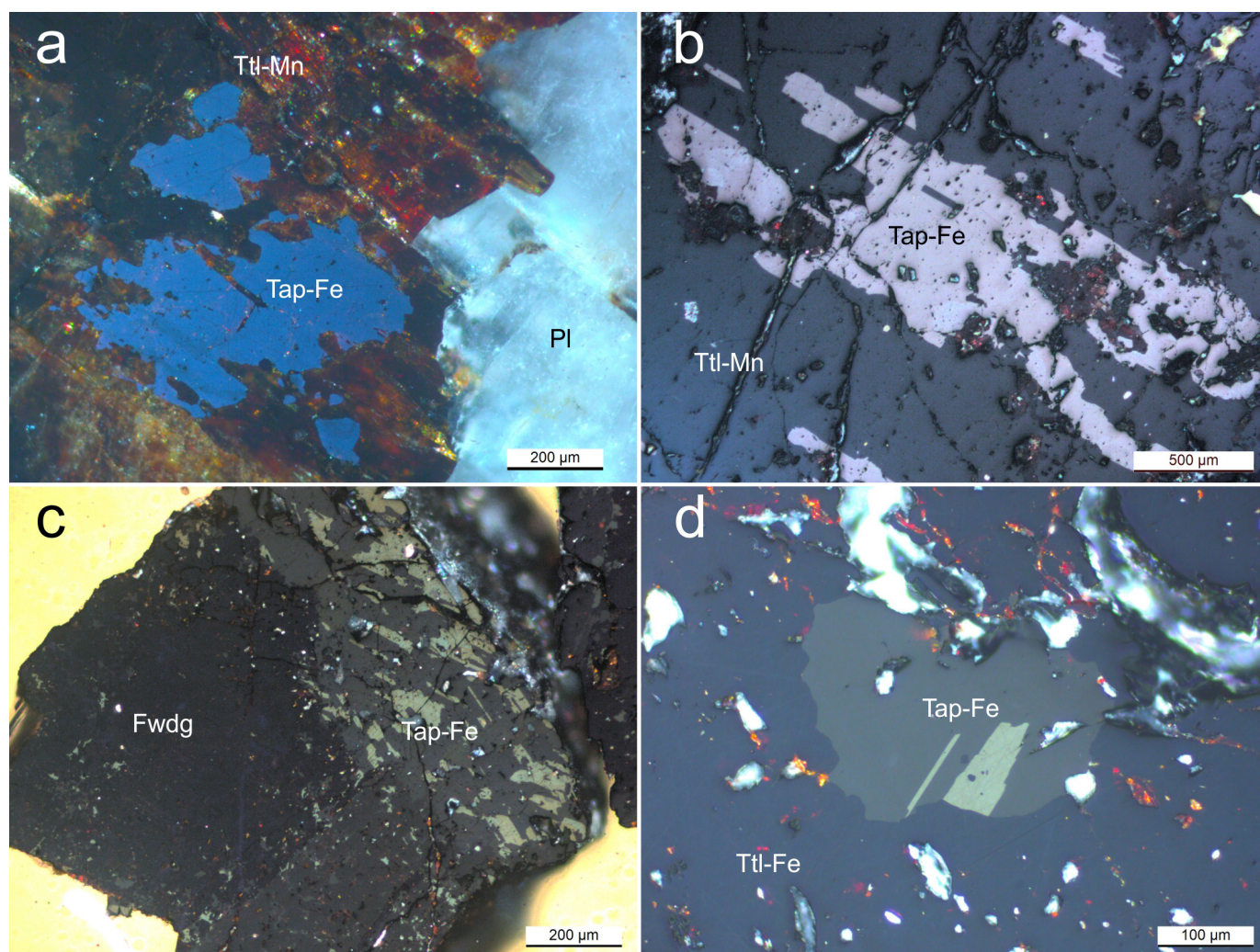


Figure 2. Photographs taken under the reflection microscope with crossed polarizers and high exposure, showing the textural relationships between tapiolite-(Fe) and the associated mineral phases. Mineral abbreviations according to IMA recommendations (Warr 2021): a) crystal of tapiolite-(Fe) from León Herido partially replaced by tantalite-(Mn); both are included in plagioclase and quartz; b) tapiolite-(Fe) crystal in optical continuity is surrounded and partially replaced by tantalite-(Mn) in a sample from the Nancy pegmatite; c) tapiolite-(Fe) showing its relationship with ferrowodginite in La Viquita pegmatite (the yellow color around the sample is because it is included in transparent epoxy resin); d) a twinned crystal of tapiolite-(Fe) from Víctor Hugo pegmatite showing replacement by tantalite-(Fe) along its margins.

(Ce), monazite-(Ce), xenotime-(Y), bertrandite, chamosite, phenakite, illite, thorite, etc. (Colombo et al. 2011).

In San Luis province, tapiolite-(Fe) occurs in several pegmatites mentioned from north to south. In the Don Pancho pegmatite it is present in the intermediate zone within an association of Qz + Ab + Spd + Ms $\pm$ (Brl + Ap) in 0.5 – 1 cm black crystals intergrown with microlite and included in albite (Vistalli 1973). In the León Herido pegmatite, tapiolite-(Fe) is associated with tantalite-(Mn) in infrequent cm-sized crystals contained in an association of Qz + Ab + Spd + Ms in the inner zones (Fig. 2a). To the south, tapiolite-(Fe) occurs in the Nancy beryl-type pegmatite as one of the HFSE-oxides associated with tantalite-(Mn), titanowodginite, ferrotitanowodginite, and hydroxycalciumicrolite in a paragenesis rarely present in the intermediate zone (Galliski et al. 2019) with ambiguous textural relationships (Fig. 2b). Tapiolite-(Fe) is fairly frequent in La Viquita, a spodumene subtype pegmatite, where it occurs as inclusions in members of the WGM and cassiterite from the core-margin assemblage. It is also present as individual subhedral crystals associated with ferrotitanowodginite (Fig. 2c), in a groundmass of muscovite, albite and quartz. It also occurs in isolated crystals in fine-grained yellow muscovite + albite from dm-sized replacement units; irregular inclusions of hafnian zircon occur sporadically. Close to this occurrence, at the San Elías pegmatite, possibly a lepidolite subtype pegmatite (Galliski et al. 1999), the mineral occurs intimately

associated with ferrowodginite and ferrotitanowodginite in a centimeter dark inclusion inside cleavelandite and quartz, found in a block sampled in the southern quarry. Tapiolite-(Fe) is present in the Víctor Hugo spodumene-subtype pegmatite where it occurs as irregular grains partially replaced (Fig. 2d), included in a 2-3 cm irregular crystal of CGM associated with microlite-group minerals. In the Cañada Honda alluvial occurrence tapiolite-(Fe) is associated in the heavy detrital fraction with CGM, monazite, scheelite, ilmenite, magnetite and garnet which provenance is related to the weathering of the pegmatites intruded in the surrounding crystalline basement (Rosello and Barbosa 1988).

METHODS

Polished sections of the samples were analyzed by electron probe micro analyzer (EPMA) with different routines in different laboratories but most of the data were obtained at the University of Manitoba, Canada. The tapiolite-(Fe) samples were analyzed in wavelength-dispersion mode in a Cameca SX50 electron microprobe, with a beam diameter of 1–2 μ m and an acceleration potential of 15 kV. A sample current of 20 nA measured on a Faraday cup and a counting time of 20 s were used for Fe, Mn, Sn, Ti, Nb, and Ta. For Mg, Ca, Sb, As, Bi, Sc, Zr, and U, 40 nA and 50 s were used.

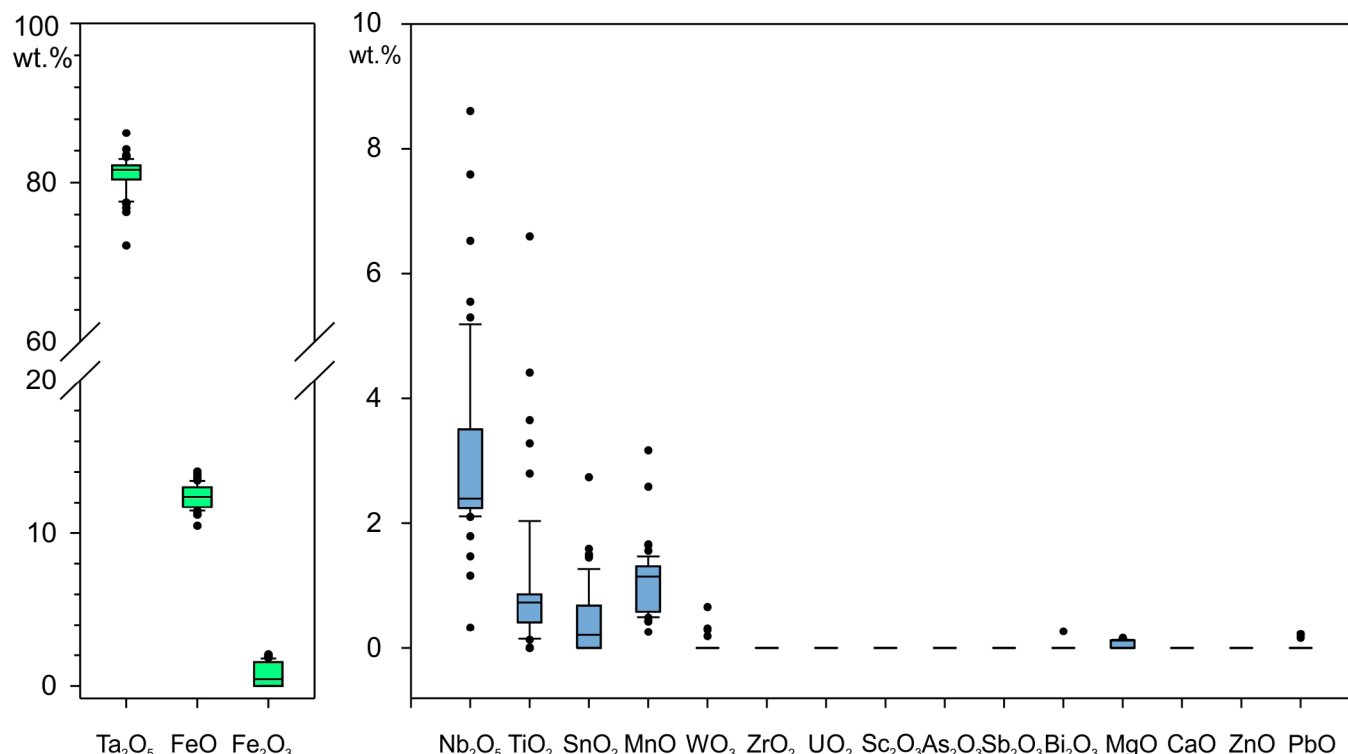


Figure 3. Bar diagram schematically showing the range, mean, and outlier compositions of major, minor, and trace element oxides of tapiolite-(Fe) from granitic pegmatites of the PPP as expressed in Table 3.

The standards and analytical lines used for the analyses were: manganotantalite (Ta, $M\alpha$), FeNb_2O_6 (Fe, $K\alpha$), MnNb_2O_6 (Nb, $L\alpha$; Mn, $K\alpha$), SnO_2 (Sn, $L\alpha$), rutile (Ti, $K\alpha$), ZrO_2 (Zr, $L\alpha$), $\text{NaScSi}_2\text{O}_6$ (Sc, $K\alpha$), YAG (Y, $L\alpha$), gahnite (Zn, $K\alpha$), MgNb_2O_6 (Mg, $K\alpha$), BiTaO_4 (Bi, $M\alpha$), CaWO_4 (W, $M\beta$), mimetite (As, $L\alpha$; Pb, $M\alpha$), CaNb_2O_6 (Ca, $K\alpha$), stibiotantalite (Sb, $L\alpha$), ThO_2 (Th glass, $M\alpha$) and UO_2 (U, $M\alpha$). Data were reduced using the PAP routine of Pouchou and Pichoir (1985).

Samples from the Nancy locality were analyzed in wavelength-dispersion mode with a Cameca SX-100 electron microprobe at the Laboratory of Electron Microscopy and Microanalysis, the joint facility of the Masaryk University and the Czech Geological Survey in Brno (Czech Republic). The following analytical conditions were applied: acceleration voltage 15 kV, beam diameter 2 μm , beam current 20 nA. The following standards and analytical lines were used: albite

(Na, $K\alpha$), $\text{CaTh(PO}_4)_2$ (Th, $M\alpha$), gahnite (Al, Zn, $K\alpha$), hematite (Fe, $K\alpha$), lammerite (As, $L\alpha$), sanidine (Si, K, $K\alpha$), MgAl_2O_4 (Mg, $K\alpha$), titanite (Ca, $K\beta$), YPO_4 (Y, $L\alpha$), Mn_2SiO_4 (Mn, $K\alpha$), vanadinite (Pb, $M\beta$), TiO_2 (Ti, $K\alpha$), zircon (Zr, $L\alpha$), Nb (Nb, $L\alpha$), CrTa_2O_6 (Ta, $M\alpha$), ScVO_4 (Sc, $K\alpha$), Sb (Sb, $L\beta$), Sn (Sn, $L\alpha$), Bi (Bi, $M\beta$), W (W, $L\alpha$) and U (U, $M\beta$). Data were reduced using the PAP routine of Pouchou and Pichoir (1985).

Averaged detection limits (3σ) were as follows (concentrations in wt.%): WO_3 (0.18), Nb_2O_5 (0.16), Ta_2O_5 (0.14), TiO_2 (0.12), SnO_2 (0.15), MgO (0.03), MnO (0.09), FeO (0.10), PbO (0.16), CaO (0.04), Sc_2O_3 (0.10), ZnO (0.15), As_2O_3 (0.12), Y_2O_3 (0.09), ZrO_2 (0.13), Sb_2O_3 (0.31), Bi_2O_3 (0.27), ThO_2 (0.11) and UO_2 (0.11).

The ratio $\text{Fe}^{2+}/\text{Fe}^{3+}$ was calculated according to stoichiometry and charge-balance considerations. The formulae were calculated on the basis of 6 atoms of oxygen

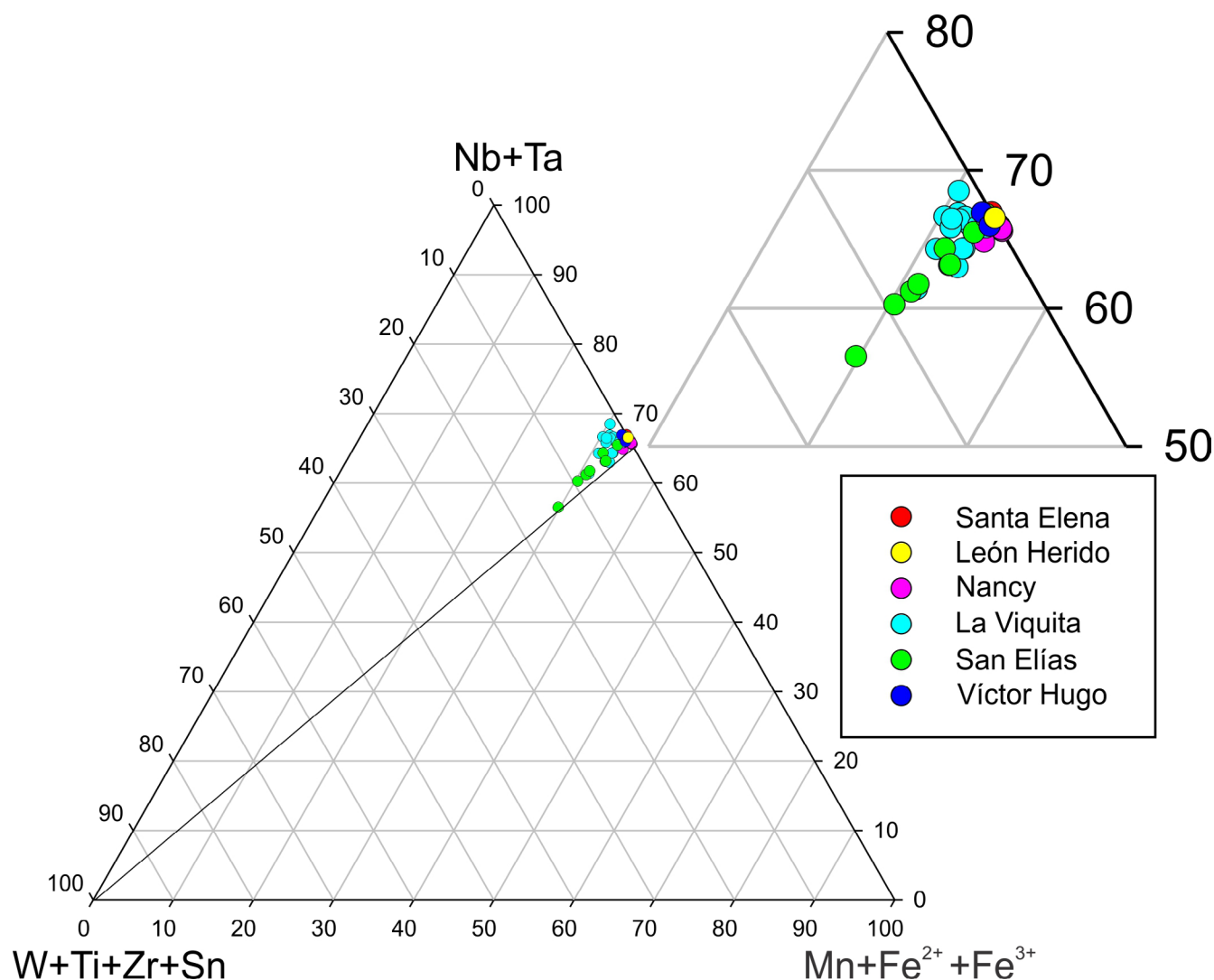


Figure 4. Compositions of tapiolite-(Fe) from different pegmatites in the $(\text{Mn} + \text{Fe}^{2+} + \text{Fe}^{3+}) - (\text{Nb} + \text{Ta}) - (\text{W} + \text{Ti} + \text{Zr} + \text{Sn})$ diagram. The 2:1 line connects the cassiterite apex with the ideal CGM composition.

Table 2. X-ray diffraction data of tapiolite-(Fe)

G19 Santa Elena					
I_{obs}	d_{obs} (Å)	d_{calc} (Å)	h	k	l
5	4.191	4.223	0	1	1
100	3.351	3.360	1	1	0
90	2.5745	2.5774	1	0	3
35	2.3684	2.3761	0	2	0
3	2.2522	2.2529	1	1	3
2	2.0742	2.0708	1	2	1
80	1.7453	1.7470	1	2	3
35	1.6772	1.6802	2	2	0
5	1.5323	1.5339	0	0	6
10	1.5004	1.5028	3	1	0
10	1.499	1.4075	3	0	3
10	1.3964	1.3954	1	1	6
3	1.2874	1.2887	2	0	6
10	1.2115	1.2110	2	3	3
2	1.1885	1.1881	0	4	0
7	1.1327	1.1328	2	2	6
5	1.1118	1.1181	2	1	7
7	1.0783	1.0790	1	4	3
7	1.0750	1.0735	3	1	6

$a = 4.752$ (4) Å / $c = 9.2034$ (5) Å / $V = 207.8$ (3) Å³

* reflections used to refine the unit-cell parameters

G74 El Peñón					
I_{obs}	d_{obs} (Å)	d_{calc} (Å)	h	k	l
10	4.558	4.595	0	0	2
12	4.215	4.215	0	1	1
100	3.3509	3.358	1	1	0
10	2.7058	2.7119	1	1	2
70	2.5745	2.5779	1	0	3
30	2.3672	2.3728	0	2	0
5	2.2718	2.2629	1	1	3
3	2.1111	2.1093	2	0	2
10	2.0652	2.0683	1	2	1
5	1.8976	1.8983	1	1	4
80	1.7485	1.7457	1	2	3
40	1.6778	1.6780	2	2	0
5	1.6544	1.6523	2	0	4
2	1.5755	1.5765	2	2	2
2	1.5605	1.5604	2	1	4
20	1.5341	1.5346	0	0	6
35	1.5009	1.5008	1	3	0
10	1.4079	1.4078	1	3	2
40	1.4057	1.4062	3	0	3
40	1.3956	1.3959	1	1	6
5	1.3036	1.3031	3	2	1
15	1.2992	1.2886	2	0	6

$a = 4.746$ (3) Å / $c = 9.2079$ (5) Å / $V = 207.4$ (1) Å³

and 3 cations. This contribution includes 38 new analyses plus 20 analyses that have already been published by Galliski et al. (2008, 2019).

The angles for identifying the crystallographic forms of the crystal from Santa Elena pegmatite were measured with an STOE optical goniometer. X-ray diffraction of the crystals from Santa Elena and El Peñón pegmatites was performed in a Debye-Scherrer camera with a diameter of 114.6 mm using CuK α radiation (1.54060 Å); intensities were visually estimated. The cell-parameter refinements of the powder diffraction data were carried out with the CELREF V3 program (Laugier and Bochu 2003).

All mineral abbreviations follow Warr (2021).

RESULTS

The crystal from the Santa Elena pegmatite shows the following forms a {100}, b {010}, p {113}, n {230}, plus another { $hk0$ } form not identified. The crystal is twinned with (b) as the twin plane that is visible on {010}, highlighted by divergent striations (Fig. 1). The X-ray diffraction diagrams of tapiolite-(Fe) from Santa Elena and El Peñón show excellent

agreement with reference data (Table 2, Appendix 1, ICDD 23-1124). The cell refinement of the Santa Elena tapiolite-(Fe) gave $a = 4.752$ (4) Å, $c = 9.2034$ (5) Å and $V = 207.8$ (3) Å³; its specific gravity measured ($n = 3$) with a Berman balance gave 7.822 (4). The cell refinement of tapiolite-(Fe) from the El Peñón pegmatite gave $a = 4.746$ (3) Å, $c = 9.2079$ (5) Å and $V = 207.4$ (1) Å³.

Electron probe microanalysis and calculated formulae are quoted in Table 3 and plotted in Figures 3, 4 and 5. The chemical composition of the studied tapiolite-(Fe) samples is fairly uniform. The ranges in wt.% of Ta₂O₅ are between 86.22 and 72.11 with a mean content of 81.59, FeO varies from 14.03 to 10.46 with a mean value of 12.38 and Fe₂O₃ from 2.06 to 0.00, with a mean of 0.44 (Table 3, Fig. 3). Minor elements (means in wt.%) are represented by: Nb (2.40 Nb₂O₅), Ti (0.74 TiO₂), Sn (0.21 SnO₂) and Mn (1.15 MnO). The trace elements have contents ≤ 0.5 % or are negligible. In terms of formula contents, the total of them reaches less than ~ 0.007 apfu. The highest value of Ta₂O₅ belongs to a León Herido tapiolite-(Fe) and the minimum to a spot in a sample from San Elías. In terms of mol % of tapiolite-(Fe), the mean value is 92, the highest 98 in a La Viquita analytical spot, and the lowest 76 in a Santa Elena spot. The analysis of tapiolite-(Fe) plot slightly

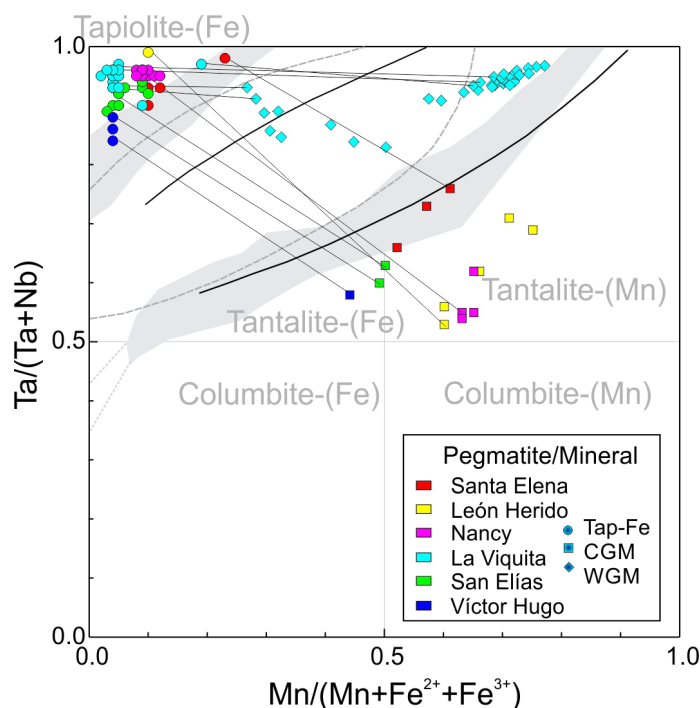


Figure 5. Chemical compositions of the tapiolite-(Fe) and the other phases (CGM and WGM) present in the same polished sections plotted in the CGM quadrilateral, with the different domains for each species. The tie lines bound the different associated minerals. The CGM and WGM compositions are extracted from Galliski and Černý (2006), and Galliski et al. (1999, 2008). The boundaries of the gap between the fields of tapiolite-(Fe) and tantalite are based on empirical observations (Černý et al. 1992, grey dashed lines), whereas the experimental boundaries at 800° C and 200 MPa (dark lines) and the natural compositional gap limited by shaded fields are from Van Lichtenvelde et al. (2018).

above the 2:1 line in the triangular compositional diagram $Mn + Fe^{2+} + Fe^{3+} - Nb + Ta - W + Ti + Zr + Sn$ (Fig. 4). In the CGM quadrilateral diagram (Fig. 5), all the analyzed points plot in the tapiolite-(Fe) compositional field.

DISCUSSION

Paragenesis and textures

Tapiolite generally appears late in the crystallization sequence of rare-element pegmatites. It is often absent in the border and wall zones and is largely restricted to the intermediate zone, the core-margin association, and the replacement units. This is the case in the studied localities, where *in situ* occurrence has been observed. Unfortunately, all tapiolite-(Fe) samples studied from the El Quemado district come from mine dump samples.

In the León Herido pegmatite, tapiolite-(Fe) occurs in the intermediate zone formed by Qz-Pl-Mc-Spd-Ms; tapiolite-(Fe) in contact with CGM is always partially replaced by tantalite-(Mn), developing caries textures and isolated relicts

of tapiolite-(Fe) that remain optically continuous. (Fig. 2a). In the Nancy pegmatite, the relationship is more ambiguous. In Figure 2b, it can be observed that anisotropic tapiolite-(Fe) crystals are partially replaced by tantalite-(Mn), leaving relicts with optical continuity. However, in other samples, tapiolite-(Fe) replaces tantalite and is in turn replaced by titanowodginite and tantalian rutile (Fig. 4c; 4e from Galliski et al. 2019). In the La Viquita pegmatite, it occurs in several forms, but in general, it is contemporaneous with or partially replaced by ferrowodginite and ferrotitanowodginite (Fig. 2c), and only cassiterite forms subsequently, generally in cavities of replacement units. In the San Elías pegmatite, it possibly forms at the same or slightly later time than ferrowodginite and ferrotitanowodginite. In contrast, the studied sample of the Víctor Hugo pegmatite shows caries contact that undoubtedly indicate that tantalite-(Fe) is replacing tapiolite-(Fe) (Fig. 4d); later, microlite was developed as a subsolidus replacement phase.

Chemical Composition

Mineral chemistry data plot in the tapiolite-(Fe) field of the CGM quadrilateral (Fig. 5) and do not differ substantially from others (e.g., Wise and Černý 1996, Van Lichtenvelde et al. 2018). In terms of $Ta\#$ ($Ta / (Ta + Nb)$) *apfu*, the highest number is that of the León Herido pegmatite (0.99) and the lowest is present in an analysis from Víctor Hugo (0.84). Santa Elena has variable values (0.90 to 0.98), and those from La Viquita, Nancy, and San Elías generally show very good clustering (averages of 0.95, 0.96, and 0.92, respectively). Comparing the results of the analysis of CGM associated with tapiolite-(Fe) (Fig. 5), the $Ta\#$ values are lower and their $Mn\#$ values higher, as usually occurs in similar pairs worldwide (Černý et al. 1992, Van Lichtenvelde et al. 2018). The differences in the contents of Fe^{3+} (calculated) between tapiolite-(Fe) and the coexisting CGM are roughly one order of magnitude larger in the first. Regarding the trace element content of tapiolite-(Fe) related to the coexisting phases: (CGM and WGM), the existing chemical data are scarce (Galliski et al. 2019, 2021) and do not allow to extract meaningful conclusions.

Origin

In all the studied occurrences of tapiolite-(Fe), this mineral forms late during the overall crystallization of the hosting pegmatites. In most of them, as it happens in the samples of Santa Elena, León Herido, San Elías, La Viquita and Víctor Hugo pegmatites, tapiolite-(Fe) crystallizes first and is followed by CGM or WGM which partially replace it. This is shown by the caries textures (Fig. 2), a classic replacement

Table 3. Chemical analyses of tapiolite-(Fe)

	SANTA ELENA					SAN ELÍAS								
	EL1A	EL1B	EL2B	EL2T	EL4BA	ELI1C	ELI1D	ELI1E	ELI2A	ELI2E	ELI2H	ELI3C	ELI3E	ELI3F
WO ₃	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Nb ₂ O ₅	3.44	5.18	4.94	3.49	1.16	5.30	3.38	3.82	5.55	4.23	4.94	3.24	3.98	3.93
Ta ₂ O ₅	80.86	78.04	77.90	81.07	83.20	76.82	80.75	77.50	72.11	77.63	77.90	82.27	80.57	81.94
TiO ₂	0.64	0.83	1.95	0.47	bdl	3.65	1.91	4.42	6.60	3.28	1.95	bdl	0.74	0.18
SnO ₂	bdl	bdl	0.49	bdl	bdl	0.68	0.28	0.61	1.07	0.55	0.49	0.36	0.27	0.15
Fe ₂ O ₃	0.50	0.47	0.75	0.51	bdl	0.21	bdl	0.17	1.67	0.23	0.75	bdl	bdl	bdl
MgO	bdl	bdl	bdl	bdl	0.03	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
MnO	1.45	1.43	0.64	1.66	3.17	0.58	1.15	1.35	0.43	0.65	0.64	1.31	0.64	0.80
FeO	12.32	12.43	13.02	12.14	10.46	13.24	12.55	12.19	12.04	13.00	13.02	12.65	13.50	13.45
PbO	0.20	bdl	bdl	0.23	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Total	99.41	98.38	99.69	99.57	98.02	100.48	100.02	100.06	99.47	99.57	99.69	99.83	99.70	100.45
W ⁶⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Nb ⁵⁺	0.130	0.194	0.181	0.132	0.045	0.189	0.125	0.137	0.192	0.154	0.181	0.123	0.149	0.147
Ta ⁵⁺	1.833	1.761	1.714	1.838	1.957	1.648	1.798	1.671	1.505	1.698	1.714	1.876	1.815	1.845
Ti ⁴⁺	0.040	0.052	0.119	0.029	0.000	0.217	0.118	0.264	0.380	0.198	0.119	0.000	0.046	0.011
Sn ⁴⁺	0.000	0.000	0.016	0.000	0.000	0.021	0.009	0.019	0.033	0.018	0.016	0.012	0.009	0.005
Fe ³⁺	0.032	0.029	0.046	0.032	0.000	0.013	0.000	0.010	0.096	0.014	0.046	0.000	0.000	0.000
Mg ²⁺	0.000	0.000	0.000	0.000	0.004	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Mn ²⁺	0.102	0.101	0.044	0.117	0.232	0.039	0.080	0.091	0.028	0.044	0.044	0.093	0.045	0.056
Fe ²⁺	0.859	0.863	0.881	0.846	0.757	0.874	0.859	0.808	0.770	0.874	0.881	0.887	0.935	0.931
Pb ²⁺	0.004	0.000	0.000	0.005	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Catsum	3.000	3.000	3.001	2.999	2.995	3.001	2.989	3.000	3.004	3.000	3.001	2.991	2.999	2.995
Oxygens	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000
Mn#	0.10	0.10	0.05	0.12	0.23	0.04	0.09	0.10	0.03	0.05	0.05	0.09	0.05	0.06
Ta#	0.93	0.90	0.90	0.93	0.98	0.90	0.93	0.92	0.89	0.92	0.90	0.94	0.92	0.93
%Tap-Fe	90	90	95	88	76	96	91	90	97	95	95	90	95	94

Formulae calculated on the basis of 6 anions and 3 cations

Shaded cells indicate previously published chemical analyses.

Ca, Sc, Zn, As, Y, Zr, Sb, Bi, Th and U were below detection limits (bdl).

texture that documents the substitution. The replacement would have occurred through magmatic crystallization under disequilibrium conditions, as it happens in the later stages of magmatic crystallization of pegmatites according to London (2008) and demonstrated in the experimental runs carried out by Van Lichtenvelde et al. (2018). The exception is the tapiolite-(Fe) from Nancy pegmatite where it replaces tantalite-(Mn) and is in turn replaced by WGM and tantalian rutile. One possible explanation is that the associated tapiolite-(Fe), ferrowodginite, ferrotitanowodginite and tantalian rutile are phases that replace the magmatic tantalite-(Mn) during a subsolidus process produced by a chemically variable hydrothermal fluid (Galliski et al. 2019).

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Table 3. Chemical analyses of tapiolite-(Fe) (cont.)

	LEÓN H.	VÍCTOR HUGO			LA VIQUITA									
	LEO1A	VH1C	VH1F	VH1G	VI11A	VI2AA	VI14A	VI8A1	VI8A2	VI8A3	VI8A4	VI8A5	VI8A6	VI8AA
WO ₃	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	0.19	0.29	bdl	bdl
Nb ₂ O ₅	0.33	8.61	6.53	7.59	4.98	1.47	1.79	2.40	2.82	3.00	2.70	2.32	2.57	3.43
Ta ₂ O ₅	86.22	76.30	79.00	77.90	78.52	84.15	83.30	81.96	82.07	81.43	80.64	81.19	81.14	77.33
TiO ₂	bdl	bdl	0.30	0.30	0.15	0.13	0.41	0.48	0.55	0.42	0.58	0.40	0.67	2.80
SnO ₂	bdl	bdl	bdl	bdl	0.93	0.27	0.44	0.70	0.85	0.79	1.50	1.02	1.49	1.59
Fe ₂ O ₃	0.30	0.12	bdl	0.42	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
MgO	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
MnO	1.46	0.64	0.50	0.64	1.17	2.59	0.72	0.59	0.54	0.56	0.26	0.58	0.49	0.54
FeO	12.44	14.03	13.66	13.88	12.63	11.27	12.82	13.07	12.54	12.74	12.30	12.44	12.60	12.75
PbO	0.16	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Total	100.91	99.70	99.99	100.73	98.38	99.88	99.48	99.20	99.37	98.94	98.17	98.24	98.96	98.44
W ⁶⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.004	0.006	0.000	0.000
Nb ⁵⁺	0.013	0.316	0.241	0.276	0.189	0.056	0.068	0.092	0.107	0.114	0.104	0.089	0.098	0.126
Ta ⁵⁺	1.981	1.682	1.756	1.703	1.791	1.937	1.916	1.880	1.875	1.868	1.864	1.883	1.859	1.715
Ti ⁴⁺	0.000	0.000	0.018	0.018	0.009	0.008	0.026	0.030	0.035	0.027	0.037	0.026	0.042	0.172
Sn ⁴⁺	0.000	0.000	0.000	0.000	0.031	0.009	0.015	0.024	0.028	0.027	0.051	0.035	0.050	0.052
Fe ³⁺	0.019	0.008	0.000	0.026	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.028
Mg ²⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Mn ²⁺	0.104	0.044	0.035	0.044	0.083	0.186	0.052	0.042	0.038	0.040	0.019	0.042	0.035	0.037
Fe ²⁺	0.879	0.951	0.934	0.933	0.886	0.798	0.907	0.922	0.881	0.899	0.874	0.887	0.888	0.869
Pb ²⁺	0.004	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Catsum	3.000	3.001	2.984	3.000	2.989	2.994	2.984	2.990	2.964	2.975	2.953	2.968	2.972	2.999
Oxygens	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000
Mn#	0.10	0.04	0.04	0.04	0.09	0.19	0.05	0.04	0.04	0.04	0.02	0.05	0.04	0.04
Ta#	0.99	0.84	0.88	0.86	0.90	0.97	0.97	0.95	0.95	0.94	0.95	0.95	0.95	0.93
%Tap-Fe	90	96	96	96	91	81	95	96	96	96	98	95	96	96

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Table 3. Chemical analyses of tapiolite-(Fe) (cont.)

	LA VIQUITA										NANCY				
	VI8B1	VI8BA	VI8BC	VI8C6	VI8C7	VI8CB	VI8CC	VI8D1	VI8D8	VI8FA	1/1	2/1	3/1	4/1	5/1
WO ₃	bdl	bdl	bdl	bdl	0.66	bdl	bdl	bdl	0.32	bdl	bdl	bdl	bdl	bdl	bdl
Nb ₂ O ₅	2.68	2.24	2.17	2.29	3.56	2.46	2.11	2.23	2.15	3.39	2.31	2.35	2.36	2.39	2.38
Ta ₂ O ₅	81.42	81.13	83.42	82.69	78.29	82.37	82.92	79.98	81.47	80.51	82.11	82.30	82.47	81.88	81.65
TiO ₂	1.03	1.67	0.44	0.30	1.57	0.20	0.20	0.53	0.58	0.86	0.94	0.85	0.80	0.79	0.85
SnO ₂	0.47	1.04	0.31	0.57	0.90	0.32	0.43	2.74	1.45	1.25	bdl	bdl	bdl	bdl	bdl
Fe ₂ O ₃	bdl	0.72	bdl	bdl	bdl	0.32	0.30	1.38	0.24	0.38	1.50	1.45	1.41	1.69	1.51
MgO	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	0.12	0.13	0.13	0.12	0.13
MnO	0.57	0.69	0.59	0.52	0.50	0.76	0.65	0.56	0.44	0.71	1.22	1.26	1.32	1.33	1.35
FeO	12.34	12.77	13.43	11.89	12.66	13.14	13.26	12.42	13.37	13.11	11.85	11.81	11.85	11.64	11.64
PbO	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Total	98.51	100.26	100.36	98.26	98.14	99.57	99.87	99.84	100.02	100.21	100.05	100.15	100.34	99.84	99.51
W ⁶⁺	0.000	0.000	0.000	0.000	0.014	0.000	0.000	0.000	0.007	0.000	0.000	0.000	0.000	0.000	0.000
Nb ⁵⁺	0.102	0.083	0.082	0.089	0.134	0.094	0.080	0.084	0.081	0.126	0.086	0.088	0.088	0.089	0.089
Ta ⁵⁺	1.868	1.811	1.896	1.925	1.778	1.884	1.895	1.805	1.848	1.803	1.844	1.845	1.850	1.843	1.844
Ti ⁴⁺	0.065	0.103	0.028	0.019	0.099	0.013	0.013	0.033	0.036	0.053	0.058	0.053	0.050	0.049	0.053
Sn ⁴⁺	0.016	0.034	0.010	0.019	0.030	0.011	0.014	0.091	0.048	0.041	0.000	0.000	0.000	0.000	0.000
Fe ³⁺	0.000	0.044	0.000	0.000	0.000	0.020	0.019	0.086	0.015	0.024	0.093	0.090	0.087	0.106	0.095
Mg ²⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.015	0.016	0.016	0.015	0.016
Mn ²⁺	0.041	0.048	0.042	0.038	0.035	0.054	0.046	0.039	0.031	0.050	0.085	0.088	0.092	0.093	0.095
Fe ²⁺	0.871	0.877	0.938	0.851	0.884	0.925	0.932	0.862	0.933	0.903	0.818	0.815	0.817	0.805	0.808
Pb ²⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Catsum	2.963	3.000	2.996	2.941	2.974	3.001	2.999	3.000	2.999	3.000	2.999	2.995	3.000	3.000	3.000
Oxygens	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000
Mn#	0.04	0.05	0.04	0.04	0.04	0.05	0.05	0.04	0.03	0.05	0.09	0.09	0.09	0.09	0.10
Ta#	0.95	0.96	0.96	0.96	0.93	0.95	0.96	0.96	0.96	0.93	0.96	0.95	0.95	0.95	0.95
%Tap-Fe	96	95	96	96	96	95	95	96	97	95	91	91	91	91	90

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Table 3. Chemical analyses of tapiolite-(Fe) (cont.)

	NANCY															
	6/1	7/1	8/1	9/1	10/1	25/1	36/1	37/1	38/1	39/1	40/1	41/1	42/1	51/1	55/1	5/1
WO ₃	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Nb ₂ O ₅	2.40	2.34	2.31	2.14	2.17	2.10	2.39	2.34	2.32	2.24	2.42	2.31	2.24	2.17	2.51	2.38
Ta ₂ O ₅	81.84	81.33	81.95	82.30	82.26	82.49	81.64	81.60	82.12	81.58	81.61	81.62	82.01	81.81	81.35	81.65
TiO ₂	0.88	0.88	0.81	0.74	0.86	0.84	0.71	0.76	0.68	0.76	0.71	0.80	0.75	0.73	0.73	0.85
SnO ₂	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Fe ₂ O ₃	1.99	1.70	1.73	1.63	1.44	1.65	1.88	1.73	1.40	1.79	2.06	1.52	1.86	1.83	1.63	1.51
MgO	0.13	0.12	0.14	0.14	0.13	0.12	0.16	0.15	0.13	0.15	0.13	0.12	0.13	0.12	0.12	0.13
MnO	1.35	1.22	1.23	1.23	1.18	1.26	1.56	1.65	1.44	1.23	1.29	1.27	1.28	1.27	1.15	1.35
FeO	11.44	11.64	11.68	11.71	11.89	11.75	11.21	11.18	11.66	11.55	11.46	11.71	11.57	11.57	11.79	11.64
PbO	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
Total	100.03	99.23	99.85	99.89	99.93	100.21	99.55	99.41	99.75	99.30	99.68	99.35	99.84	99.50	99.28	99.51
W ⁶⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Nb ⁵⁺	0.089	0.088	0.086	0.080	0.081	0.078	0.090	0.088	0.087	0.084	0.091	0.087	0.084	0.082	0.094	0.089
Ta ⁵⁺	1.832	1.840	1.844	1.854	1.853	1.853	1.842	1.844	1.855	1.847	1.837	1.848	1.846	1.850	1.841	1.844
Ti ⁴⁺	0.054	0.055	0.050	0.046	0.054	0.052	0.044	0.048	0.042	0.048	0.044	0.050	0.047	0.046	0.046	0.053
Sn ⁴⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Fe ³⁺	0.123	0.106	0.108	0.102	0.090	0.102	0.117	0.108	0.088	0.112	0.128	0.095	0.116	0.114	0.102	0.095
Mg ²⁺	0.016	0.015	0.017	0.017	0.016	0.015	0.020	0.019	0.016	0.019	0.016	0.015	0.016	0.015	0.015	0.016
Mn ²⁺	0.094	0.086	0.086	0.086	0.083	0.088	0.110	0.116	0.101	0.087	0.090	0.090	0.090	0.089	0.081	0.095
Fe ²⁺	0.788	0.810	0.808	0.811	0.824	0.811	0.778	0.777	0.810	0.804	0.793	0.815	0.801	0.804	0.821	0.808
Pb ²⁺	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Catsum	2.996	3.000	2.999	2.996	3.001	2.999	3.001	3.000	2.999	3.001	2.999	3.000	3.000	3.000	3.000	3.000
Oxygens	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000	6.000
Mn#	0.09	0.09	0.09	0.09	0.08	0.09	0.11	0.12	0.10	0.09	0.09	0.09	0.09	0.09	0.08	0.10
Ta#	0.95	0.95	0.96	0.96	0.96	0.96	0.95	0.95	0.96	0.96	0.95	0.96	0.96	0.96	0.95	0.95
%Tap-Fe	91	91	91	91	92	91	89	88	90	91	91	91	91	91	92	90

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