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TARBAGATAITE, $(K,\square)_2(Ca,Na)(Fe^{2+},Mn)_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH,F)$, A NEW ASTROPHYLLITE-GROUP MINERAL SPECIES FROM THE VERKHNEE ESPE DEPOSIT, AKJAILYAUTAS MOUNTAINS, KAZAKHSTAN: DESCRIPTION AND CRYSTAL STRUCTURE

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Abstract

Tarbagataite, $(K,\square)_2(Ca,Na)(Fe^{2+},Mn)_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH,F)$, is a new Ca- and Fe^{2+}-dominant astrophyllite-group mineral discovered in a pegmatite in the Verkhnee Espe deposit, Akjailyautas Mountains, Kazakhstan. Tarbagataite occurs as intimate intergrowths of tarbagataite and astrophyllite flakes. Dimensions of these intergrowths range up to 10 × 3 × 0.2 mm. Flakes are elastic, brown or pale golden brown, with a colorless to very pale yellow streak and a vitreous to pearly lustre. Tarbagataite is opaque in large grains, transparent in thin flakes, with a Mohs hardness of 3, and does not fluoresce under cathode or ultraviolet light. Cleavage is perfect parallel to {001} and moderate parallel to {010}; no parting was observed. Its calculated density is 3.263 g/cm³. The mineral is biaxial positive with α 1.710, β 1.715, γ 1.745 (all ± 0.003, λ 589 nm), $2V_{\mathrm{meas.}} = 37(3)°$, $2V_{\mathrm{calc.}} = 45°$. It is pleochroic according to the scheme $X < Z < Y$, where X = yellow brown, Y = orange red, Z = yellow orange. Tarbagataite is triclinic, space group $P\bar{1}$, a 5.3863(3), b 11.9141(6), c 11.7171(6) Å, α 112.978(2), β 94.641(2), γ 103.189(2)°, V 661.84(9) Å³, $Z = 1$. The strongest lines in the X-ray powder-diffraction pattern [d(Å)(I)(hkl)] are: 3.258(100)($\bar{1}\bar{1}3$), 4.095(80)(021), 2.858(80)($0\bar{1}4$), 2.761(70)($1\bar{4}2,\bar{1}31$), 3.497(50)(030), 2.560(50)($130,1\bar{4}3$). 3.735(30)($0\bar{2}3$), 2.646(30)($\bar{2}11,004$), 3.005(20)(013). Chemical analysis by electron microprobe gave Nb_2O_5 2.98, SnO_2 1.20, ZrO_2 0.32, TiO_2 9.29, SiO_2 36.11, Al_2O_3 0.12, ZnO 0.12, FeO 18.71, MnO 15.48, CaO 2.58, MgO 0.83, Cs_2O 0.38, Rb_2O 1.28, K_2O 2.67, Na_2O 1.14, F 0.49, H_2O 3.11, O=F –0.21, sum 96.60 wt.%; H_2O was determined from structure refinement, OH + F = 5 *apfu*. The empirical formula, calculated on 31 anions (O + F) pfu, is $(K_{0.76}Rb_{0.18}Na_{0.12}Cs_{0.04}\square_{0.90})_{\Sigma 2}(Ca_{0.62}Na_{0.38})_{\Sigma 1}(Fe^{2+}_{3.51}Mn_{2.94}Mg_{0.28}Zr_{0.02}Zn_{0.02}\square_{0.23})_{\Sigma 7}(Ti_{1.57}Nb_{0.30}Sn_{0.11}Zr_{0.02})_{\Sigma 2}(Si_{8.09}Al_{0.03})_{\Sigma 8.12}O_{30.65}H_{4.65}F_{0.35}$, $Z = 1$. The simplified and endmember formulae are $(K,\square)_2(Ca,Na)(Fe^{2+},Mn)_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH,F)$ and $(K\square)CaFe^{2+}_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH)$, respectively. The infrared spectrum of the mineral contains the following absorption bands: 455, 531, 570, 656, 698, 950 with shoulders at 1078 and 1064, 1637, 3656 to 3277 cm^{-1}. The crystal structure of tarbagataite was refined to an R_1 index of 5.82 %. Tarbagataite is isostructural with astrophyllite, ideally $K_2NaFe^{2+}_7Ti_2(Si_4O_{12})_2O_2(OH)_4F$. Tarbagataite differs from astrophyllite in the composition of the interstitial A and B sites and the X site: $(K,\square)_2$ (A), (Ca,Na) (B), and (OH,F) (X). Tarbagataite and astrophyllite are related by the substitution ${}^{A}\square + {}^{B}Ca^{2+} + {}^{X}(OH)^{-} \leftrightarrow {}^{A}K^{+} + {}^{B}Na^{+} + {}^{X}F^{-}$. The name is for the locality where the mineral was discovered: the Verkhnee Espe deposit is located in the northern part of the Tarbagatai mountain range in the Akjailyautas Mountains of Kazakhstan.

Keywords: tarbagataite, new mineral species, chemical analysis, crystal structure, astrophyllite group, the Verkhnee Espe deposit, Akjailyautas Mountains, Kazakhstan.

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Introduction

Astrophyllite, ideally $K_2NaFe^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4F$, from the northern offshoots of the Tarbagatai mountain range in the Akjailyautas Mountains, Kazakhstan, has been studied since the 1950s, when the rare-metal Verkhnee Espe deposit was discovered. Spatially and genetically related rare-metal mineralization is confined to two small oval-shaped outcrops (Large and Small) of an intrusion of alkali granite in the northern exocontact of the Akjailyautas granite massif (Stepanov & Bekenova 2009). Astrophyllite occurs in numerous small concentrations of rare-metal mineralization associated with pegmatitic features, the granites themselves, and fenites above the domed exocontact in shear zones in the host rocks. In the 1970s-1980s A.V. Stepanov found splitting of several reflections in X-ray powder patterns of astrophyllite from the Verkhnee Espe deposit and suggested an intergrowth of astrophyllite and two or more minerals of the astrophyllite group which could not be distinguished visually. The composition of astrophyllite at the Verkhnee Espe deposit varies greatly, with a wide range in minor elements, *e.g.*, Li, Rb, Cs, Be, Zn, Zr, Sn, Pb, Mg, Al, Nb, and V. Stepanov *et al.* (2008) reported quantitative patterns of substitution: Mn, Zn, Mg, Pb, and Li for Fe; Nb, Zr, Sn, and V for Ti; Na, Ca, Cs and Rb for K. Tarbagataite was discovered during a systematic analytical study of astrophyllite.

Tarbagataite is isostructural with astrophyllite, ideally $K_2NaFe^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4F$ (Piilonen *et al.* 2003b); niobophyllite, ideally $K_2NaFe^{2+}{}_7(Nb,Ti)_2(Si_4O_{12})_2O_2(OH)_4(O,OH)$ (Cámara *et al.* 2010); nalivkinite, ideally $Li_2NaFe^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4F$ (Uvarova *et al.* 2008); kupletskite, ideally $K_2NaMn^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4F$ (Piilonen *et al.* 2001); kupletskite-(Cs), ideally $Cs_2NaMn^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4F$ (Cámara *et al.* 2010); niobokupletskite, $K_2NaMn^{2+}{}_7(Nb,Ti)_2(Si_4O_{12})_2O_2(OH)_4(O,OH)$ (Piilonen *et al.* 2000); and zircophyllite, ideally $K_2NaMn^{2+}{}_7Zr_2(Si_4O_{12})_2O_2(OH)_4F$ (Kapustin 1973); the structure reference is given, except for zircophylite, for which the structure has not yet been refined. The stoichiometries of the above astrophyllite-group minerals and magnesioastrophyllite, ideally $K_2Na(Fe^{2+}{}_4Mg_2Na)Ti_2(Si_4O_{12})_2O_2(OH)_4$, can be described by a general formula of the form $A_2BC_7D_2(T_4O_{12})_2O_2(OH)_4X_{0-1}$ (Cámara *et al.* 2010, modified after Piilonen *et al.* 2003a) where $^{[5-13]}A$ = K, Li, Rb, Cs, Na, H_2O or $\square$; $^{[10]}B$ = Na or Ca; $^{[6]}C$-group cations at the M(1-4) sites = Fe^{2+}, Mn, Fe^{3+}, Na, Mg or Zn; $^{[5,6]}D$ = Ti, Nb, Zr or Sn; $^{[4]}T$ = Si or Al and X = F, OH, O or $\square$.

The name is for the locality where the mineral was discovered; the Verkhnee Espe deposit is located in the northern part of the Tarbagatai mountain range. The new mineral species and its name have been approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association (IMA 2010-048). The holotype specimen of tarbagataite is deposited at the Geological Museum of the Satpaev Institute of Geological Sciences, ul. Kabanbai batyr, 69, Almaty, 050010, Kazakhstan, catalogue No. 3009/2010.

Occurrence And Associated Minerals

Samples containing tarbagataite-astrophyllite intergrowths were taken from a pegmatitic quartz-feldspar vein several hundred meters from outcrops of alkaline granite. The vein ranges in thickness from 10 to 120 cm and crosscuts the layering in the Lower Carboniferous country rock. The latter consists of tuff, siltstone, sandstone, shales and rare interbedded carbonaceous sandstone (containing crinoids and other plant fossils), all intruded by basic dikes. There is a shear zone that was the conduit for fluids carrying rare elements, probably together with F, Cl, $B(OH)_3$, CO_2, and H_2O. Minerals show extremely strong zoning, both on a grain-to-grain scale and within individual grains, suggesting rapid change in the composition of the mineralizing fluids; there is no well-developed systematic metasomatic zoning.

Astrophyllite is prominent throughout the quartz-feldspar vein as streaks and segregations up to 10 cm thick, forming up to 20% by volume of the rock. Tarbagataite and other minerals of astrophyllite group (kupletskite, niobophyllite) occur where the vein crosses a basic dike with a thickness of about 5 m. Astrophyllite and tarbagataite are associated with microcline, albite, quartz, aegirine, kupletskite, zircon, thorite, xenotime, fergusonite, fersmite, euxenite-(Y), ilmenite, columbite-(Mn), zinc pyrophanite, keiviite, thalenite-(Y), cappelenite-(Y), eudialyte, jinshajiangite, milarite, helvite, bertrandite, and bavenite, among others.

Physical and Optical Properties

The main properties of tarbagataite are presented in Table 1, where they are compared to those of astrophyllite. Tarbagataite is intimately associated with astrophyllite as lamellar intergrowths (Fig. 1) up to 10 × 3 × 0.2 mm. Discrete layers of tarbagataite seldom exceed 0.03 mm in thickness. In polished thin section there are subtle differences in hardness and reflectivity between tarbagataite and astrophyllite. Tarbagataite is brown to pale golden brown; its streak is colorless with a hint of yellow, and its lustre varies from vitreous to pearly. It is opaque in large grains and transparent in thin flakes and is non-fluorescent under 240-400 nm ultraviolet radiation. Thin flakes are elastic. Tarbagataite has a perfect cleavage parallel to {001} and moderate cleavage parallel to {010}, and a Mohs hardness of 3; fracture and parting were not observed. The density of the mineral could not be measured accurately due to a lack of suitable material. Its calculated density (using the empirical formula) is 3.263 g/cm^3. The mineral is biaxial positive with α 1.710, β 1.715, γ 1.745 (all ± 0.003, λ 589 nm), $2V_{\text{meas.}} = 37(3)°$, $2V_{\text{calc.}} = 45°$. We tried to determine dispersion by measuring extinction angles on a spindle stage at two different wavelengths of light (589 and 432 nm). Extinction of tarbagataite could not be determined at 432 nm owing to excessive absorption of light, and thus the dispersion could not be determined. Tarbagataite is pleochroic, $X < Z < Y$, where X = yellow brown, Y = orange red, Z = yellow orange (λ 589 nm). The optical orientation is given in Table 2. A Gladstone-Dale calculation gives a compatibility index of 0.004, which is rated as superior (Mandarino 1981).

TABLE 1. COMPARISON OF TARBAGATAITE AND ASTROPHYLLITE

	tarbagataite	astrophyllite*
Endmember formula	$(K\square)CaFe^{2+}{}_7Ti_2$ $(Si_4O_{12})_2O_2(OH)_5$	$K_2NaFe^{2+}{}_7Ti_2$ $(Si_4O_{12})_2O_2(OH)_4F$
System	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	5.3863(3)	5.36-5.42
b	11.9141(6)	11.85-11.95
C	11.7171(6)	11.66-11.75
α (°)	112.978(2)	112.03-113.13
Β	94.641(2)	94.52-94.64
Γ	103.189(2)	103.08-103.14
V (Å^3)	661.84(9)	652.9-668.5
Z	1	1
D_{meas}/D_{calc} (g/cm^3)	n.d./3.263	3.09-3.23/3.35
Strongest lines in the powder pattern d_{meas} (Å) (*I*)	3.258 (100) 4.095 (80) 2.858 (80) 2.761 (70) 3.497 (50) 2.560 (50)	10.6 (100) 3.51 (80) 2.77 (60) 2.64 (60) 2.57 (60) 1.76 (30)
Optical character	biaxial (+)	biaxial (+)
n_α	1.710	1.678-1.695
n_β	1.715	1.703-1.726
n_γ	1.745	1.733-1.758
n_α–n_γ	0.035	0.025-0.063
$2V_{meas}$ (°)	37 (3)	66-84
Color	Brown	Bronze yellow to golden yellow, brown to reddish brown
Pleochroism	*X* = yellow brown *Y* = orange red *Z* = yellow orange	*X* = deep orange red *Y* = orange yellow *Z* = lemon yellow

*Data from Anthony *et al*. (1995) except for cell parameters (Piilonen *et al*. 2003b) and the optical orientation of astrophyllite (Nickel *et al.* 1964).

TABLE 2. OPTICAL ORIENTATION (°) FOR TARBAGATAITE

	a	b	c
X	89.5	165.6	58.5
Y	94.8	95.4	147.0
Z	4.8	103.3	98.9

The powder IR spectrum was recorded with a Nicolet FTIR 740 spectrophotometer in the range 4000-400 cm^{-1} (Fig. 2). In the principal OH-stretching region (3800-3000 cm^{-1}), the spectrum shows several sharp bands between 3656 and 3277cm^{-1} in accordance with the presence of several OH groups in the structure. There is a very weak band at 1637 cm^{-1} due to the H–O–H bend of H_2O groups adsorbed on the sample, and weak bands around 1436 cm^{-1} possibly due to (inclusions of) a carbonate mineral. There is a sharp intense band at 950 cm^{-1} with shoulders at 1078 and 1064 cm^{-1} due to various stretching modes of the SiO_4 group, and weaker bands at 698, 656, 570, 531, and 455 cm^{-1} due to complex lattice modes involving the octahedra and coupled motions between the various polyhedra.

CHEMICAL COMPOSITION

The chemical composition of tarbagataite (Table 3) was determined with a Cameca SX-100 electron microprobe operating in wavelength-dispersion mode with an accelerating voltage of 15 kV, a specimen current of 20 nA, a beam size of 10 µm and count times on peak and background of 20 and 10 s, respectively. We used the following standards: $Ba_2NaNb_5O_{15}$ (Nb), titanite (Ti), SnO_2 (Sn), zircon (Zr), diopside (Si, Ca), andalusite (Al), fayalite (Fe), spessartine (Mn), forsterite (Mg), gahnite (Zn), $SrTiO_3$ (Sr), pollucite (Cs), orthoclase (K), albite (Na), and F-bearing riebeckite (F); Cr, Ba, Ta, and Pb were sought but not detected. Lithium and Be were sought using LA-ICP-MS but

TABLE 3. CHEMICAL COMPOSITION AND UNIT FORMULA FOR TARBAGATAITE

Constituent	wt.%	Range	Element	*apfu*
Nb_2O_5	2.98	2.64 – 3.59	Nb	0.30
SnO_2	1.20	1.11 – 1.29	Sn	0.11
ZrO_2	0.32	0.29 – 0.36	Zr	0.04
TiO_2	9.29	8.89 – 9.53	Ti	1.57
SiO_2	36.11	35.83 – 36.30	Si	8.09
Al_2O_3	0.12	0.09 – 0.14	Al	0.03
ZnO	0.12	0.06 – 0.16	Zn	0.02
FeO	18.71	18.09 – 19.49*	Fe^{2+}	3.51
MnO	15.48	14.71 – 16.11	Mn	2.94
CaO	2.58	2.47 – 2.77	Ca	0.62
MgO	0.83	0.76 – 0.93	Mg	0.28
Cs_2O	0.38	0.35 – 0.43	Cs	0.04
Rb_2O	1.28	1.00 – 1.46	Rb	0.18
K_2O	2.67	2.45 – 2.88	K	0.76
Na_2O	1.14	0.90 – 1.28	Na	0.50
F	0.49	0.32 – 0.78	Σ cations	18.99
H_2O*	3.11		F	0.35
O = F	–0.21		Σ(O + F)	31.00
Total	96.60		H	4.65

*calculated from structure refinement, OH + F = 5 *apfu*.

not detected. The amount of H_2O was not determined directly because of insufficient material; it was calculated by stoichiometry from the crystal-structure analysis and its presence was confirmed by infrared spectroscopy. The empirical formula for tarbagataite calculated on 31 anions (O + F) per formula unit ($Z = 1$) is as follows: $(K_{0.76}Rb_{0.18}Na_{0.12}Cs_{0.04}\square_{0.90})_{\Sigma 2}(Ca_{0.62}Na_{0.38})_{\Sigma 1}(Fe^{2+}_{3.51}Mn_{2.94}Mg_{0.28}Zr_{0.02}Zn_{0.02}\square_{0.23})_{\Sigma 7}$ $(Ti_{1.57}Nb_{0.30}Sn_{0.11}Zr_{0.02})_{\Sigma 2}(Si_{8.09}Al_{0.03})_{\Sigma 8.12}O_{30.65}H_{4.65}F_{0.35}$; the amount of H_2O was determined from structure refinement: OH + F = 5 *apfu*.

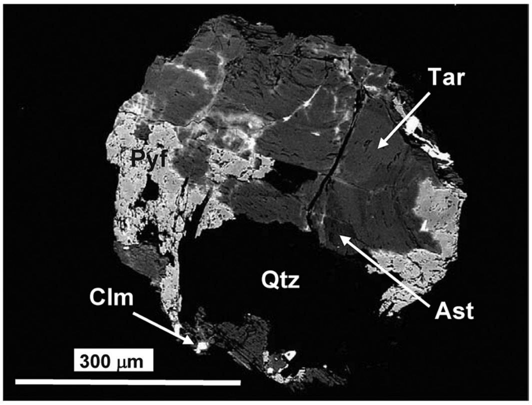

FIG. 1. BSE image of tarbagataite (Tar, grey) intergrown with astrophyllite (Ast, dark grey), zinc pyrophanite (Pyf, light grey), columbite-(Mn) (Clm, white), and quartz (Qtz, black) (polished surface of a thin section).

X-RAY POWDER DIFFRACTION

The X-ray powder diffraction data for tarbagataite were collected using a Debye-Scherrer camera 57.3 mm in diameter. Table 4 shows the X-ray powder diffraction data (for Co*K*α, λ = 1.79026 Å) together with the refined unit-cell dimensions; the latter are in close agreement with corresponding values determined by single-crystal diffraction (Table 1).

CRYSTAL STRUCTURE

X-ray data collection and structure refinement

Single-crystal X-ray data for tarbagataite were collected with a Bruker AXS SMART APEX diffractometer with a CCD detector (Mo*K*α radiation). The intensities of X-ray diffraction reflections were collected to 60.00° 2θ using 30 s per 0.5° frame and an empirical absorption correction (SADABS, Sheldrick 2008) was applied. As there were hardly any reflections at high angles, the maximum 2θ limit for X-ray diffraction intensities was cut to 54.99° 2θ, corresponding to 7434 reflections with $-6 < h < 6$, $-15 < k < 15$, $-15 < l < 15$. The refined unit-cell parameters were obtained from 9295 reflections with $I > 10\sigma I$ (Tables 1, 5). We used atom coordinates of Sn-rich astrophyllite (space group $P\overline{1}$, Cámara *et al.* 2010) as a starting model; with these, the crystal structure of tarbagataite was refined to an R_1 value of 5.82% with the Bruker SHELXTL version 5.1 system of programs (Sheldrick 2008). For tarbagataite, we observed splitting of the *A* site

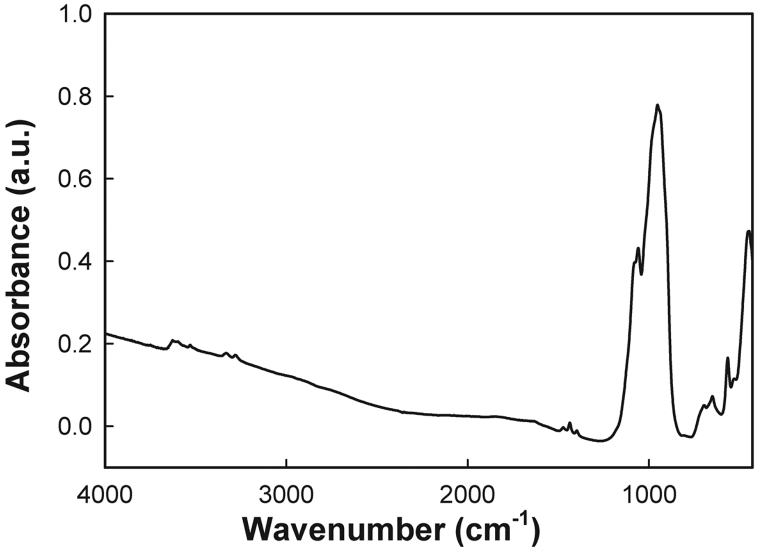

FIG. 2. IR spectrum of tarbagataite.

into two sites, *A*(1) and *A*(2), ~50 and 6% occupied by K and Na, respectively, and separated by a short distance (0.44 Å). Site-scattering values were refined for the *D* site (scattering curve of Ti), *M* sites (scattering curve of Fe), *A* site (scattering curve of K), and *B* site (scattering curve of Ca). Scattering curves for neutral atoms were taken from the International Tables for Crystallography (Wilson 1992). Hydrogen atoms were included at the final stages of refinement, and both H positions were softly constrained by setting the O–H distances equal to 0.98(1) Å. Details of data collection and structure refinement are given in Table 5, final atom parameters are given in Table 6, selected interatomic distances in Table 7, refined site-scattering values and assigned populations for selected sites are given in Table 8, details of hydrogen bonding in Table 9, and bond valences for selected anion sites in Table 10. Observed and calculated structure-factors and anisotropic displacement parameters may be obtained from The Depository of Unpublished Data on the MAC website [document Tarbagataite CM50_159].

Topology of the structure

Tarbagataite is isostructural with astrophyllite. In the astrophyllite structure, the main structural unit is an HOH layer (Fig. 3). The M(1), M(2), M(3), and M(4) octahedra (C group) share edges to form an O sheet of close-packed octahedra. The (T_4O_{12}) astrophyllite ribbons (Belov 1976) share common vertices with D octahedra to form the heteropolyhedral (H) sheet. In the crystal structure of astrophyllite, the O sheet and two H sheets form an HOH layer of composition

TABLE 4. X-RAY POWDER DIFFRACTION DATA FOR TARBAGATAITE

$I_{obs.}$	$d_{obs.}$(Å)	$d_{calc.}$ (Å)	*h*	*k*	*l*
<10	6.992	6.276	0	1	1
10	4.378	4.368	$\bar{1}$	$\bar{1}$	1
80	4.095	4.084	0	2	1
30	3.735	3.747	0	$\bar{2}$	3
50	3.497	3.509	0	3	0
100*	3.258	3.259	$\bar{1}$	$\bar{1}$	3
20d	3.005	2.996	0	1	3
80	2.858	2.871	0	$\bar{1}$	4
70	2.761	2.774	1	$\bar{4}$	2
		2.761	$\bar{1}$	$\bar{3}$	1
30	2.646	2.644	$\bar{2}$	1	1
		2.651	0	0	4
50	2.560	2.565	1	3	0
		2.568	1	$\bar{4}$	3
10	2.298	2.291	2	$\bar{1}$	2
		2.291	1	3	1
		2.287	1	$\bar{4}$	4
<10	1.762	1.765	1	3	3
		1.758	1	$\bar{4}$	6
<10	1.638	1.637	0	$\bar{7}$	2
		1.633	0	$\bar{7}$	4
		1.638	$\bar{1}$	4	4
<10	1.563	1.564	$\bar{3}$	$\bar{2}$	2
		1.562	$\bar{3}$	5	0

*Diffuse.

Indexed on a = 5.350(8), b = 11.911(16), c = 11.716(18) Å, α = 112.61(11), β = 94.90(18), γ = 103.48(12)°, V = 657.4(11) Å^3; d_{calc} and *hkl* values are from the powder pattern calculated from single-crystal data.

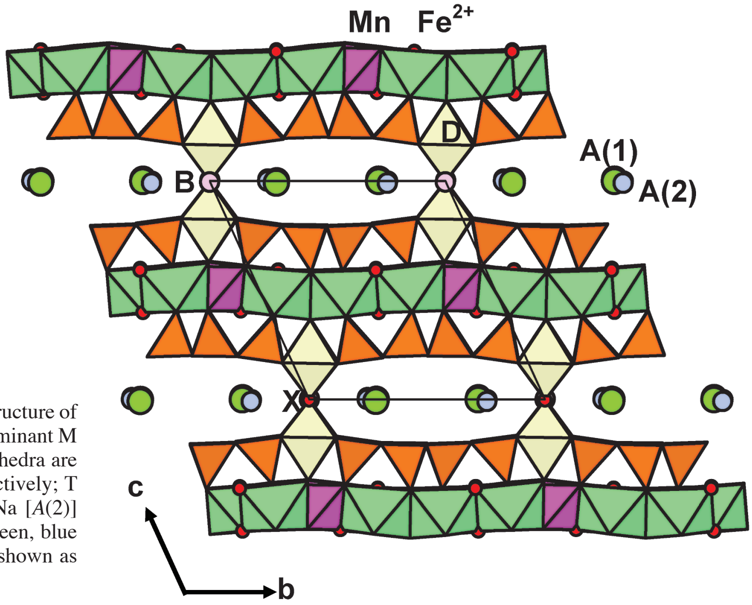

FIG. 3. General view of the crystal structure of tarbagataite. The Mn- and Fe^{2+}-dominant M octahedra and Ti-dominant D octahedra are magenta, green and yellow, respectively; T tetrahedra are orange, K [*A*(1)], Na [*A*(2)] and Ca (*B*) atoms are shown as green, blue and pink spheres, OH groups are shown as small red spheres.

$C_7D_2(T_4O_{12})_2O_2(OH)_4X$, where C_7 represents cations at the *M*(1-4) sites of the O sheet. Along [001], the HOH layers connect *via* common X anions of D octahedra and interstitial cations at the *A* and *B* sites (Fig. 3). In the tarbagataite structure, labelling of the anion and cation sites is identical to that of astrophyllite (Piilonen *et al*. 2003b), in accord with the general formula $A_2BC_7D_2(T_4O_{12})_2O_2(OH)_4X_{0\text{-}1}$ (see above).

Cation sites

Site populations were assigned on the basis of the refined site-scattering values (Table 8), the chemical formula (Table 3) derived from electron-microprobe data, and the observed mean bond-lengths (Table 7).

There are eleven cation sites in the crystal structure of tarbagataite.

M sites of the C group in the O sheet: In tarbagataite the sum of the medium-sized divalent $(Fe^{2+}_{3.51}Mn_{2.94}Mg_{0.28}Zn_{0.02})$ and tetravalent $(Zr^{4+}_{0.02})$ cations is 6.79 *apfu*, with a vacancy of 0.23 *pfu* (Table 3) and a total calculated scattering of 169.52 *epfu* (electrons per formula unit). For the *M*(1-4) sites the total refined site-scattering is 168.4 *epfu* (Table 8). In astrophyllite-type structure the sizes of the M octahedra follow the pattern M(1) > M(2) > M(3) > M(4) due to different linkages of individual M octahedra and polyhedra of the H sheets (Sokolova 2012). In tarbagataite <M(1)-–O> = 2.185 Å, <M(2)–O> = 2.170 Å, <M(3)–O> = 2.153 Å, and <M(4)–O> = 2.126 Å (Tables 7, 8). In accord with the sizes of the M octahedra, we assign smaller cations Mg [*r* = 0.72 Å; Shannon (1976)], Zr (*r* = 0.72 Å) and Zn (*r* = 0.74 Å) to the *M*(3,4) sites, and a significant amount of larger Mn (*r* = 0.83 Å) to the *M*(1,2) sites; in accordance with individual site-scattering values, we distribute 0.23 □ *pfu* over the *M*(1-3) sites. Note that Mn and Fe^{2+} are the dominant cations at the *M*(1) and *M*(2-4) sites, respectively (Table 8). The M(1) and M(2-4) atoms are coordinated by five O atoms and one OH group and four O atoms and two OH groups, respectively (Tables 7, 8). The *M*(1-4) sites give $(Fe^{2+}_{3.51}Mn_{2.94}Mg_{0.28}Zr_{0.02}Zn_{0.02}\square_{0.23})$ *pfu* (total charge 13.58^+), which is in close agreement with the simplified and endmember compositions of $(Fe^{2+},Mn^{2+})_7$ and Fe^{2+}_7 *apfu* (charge 14^+).

H sheet: There are five cation sites in the H sheet. The *D* site is occupied by $Ti_{1.57}Nb_{0.30}Sn_{0.11}Zr_{0.02}$ *apfu* and is coordinated by five O atoms and an X anion where X = OH, F; <D–ϕ> is equal to 1.957 Å (ϕ:

TABLE 5. MISCELLANEOUS REFINEMENT DATA FOR TARBAGATAITE

a (Å)	5.3868(3)
b	11.9141(6)
c	11.7171(6)
α (°)	112.978(2)
β	94.641(2)
γ	103.189(2)
V (Å^3)	661.84(9)
Space group	$P\bar{1}$
Z	1
Absorption coefficient (mm^{-1})	5.06
F(000)	630.1
$D_{calc.}$ (g/cm^3)	3.263
Crystal size (mm)	0.020 x 0.300 x 0.300
Radiation/filter	MoKα/graphite
2θ-range for structure refinement (°)	54.99
R(int) (%)	2.26
Reflections collected	7434
Independent reflections	2822
$F_o > 4\sigma F$	3031
Refinement method	Full-matrix least squares on F^2, fixed weights proportional to $1/\sigma F_o{}^2$
No. of refined parameters	250
Final $R_{(obs)}$ (%)	
$[F_o > 4\sigma F]$	5.82
R_1	6.27
wR_2	13.51
Goodness of fit on F^2	1.266

TABLE 6. ATOM POSITIONS AND EQUIVALENT* DISPLACEMENT PARAMETERS FOR TARBAGATAITE

Atom	*x*	*y*	*z*	U_{eq} (Å^2)
M(1)	0.85010(17)	0.20477(8)	0.47800(9)	0.0111(3)
M(2)	0.27798(17)	0.06692(8)	0.48669(9)	0.0114(3)
M(3)	0.42099(17)	0.35140(8)	0.48311(9)	0.0118(3)
M(4)	0	½	½	0.0098(5)
D	0.07770(16)	0.08463(8)	0.19305(9)	0.0077(3)
T(1)	0.6809(3)	0.27333(14)	0.23257(15)	0.0092(4)
T(2)	0.8141(3)	0.54771(15)	0.25587(16)	0.0113(4)
T(3)	0.3797(3)	0.67619(15)	0.25908(16)	0.0123(4)
T(4)	0.5060(3)	0.93184(15)	0.23728(15)	0.0091(4)
A(1)	0.1453(8)	0.2810(3)	0.9949(4)	0.0322(8)
A(2)	0.1284(5)	0.247(5)	1.003(5)	0.0322(8)
B	½	0	0	0.0199(5)
O(1)	0.7280(8)	0.3192(4)	0.3844(4)	0.0120(8)
O(2)	0.1468(8)	0.1601(4)	0.3686(4)	0.0148(9)
O(3)	0.1277(8)	0.3914(4)	0.5919(4)	0.0128(9)
O(4)	0.2985(9)	0.4648(4)	0.4023(4)	0.0152(9)
O(5)	0.9928(9)	0.1181(4)	0.5921(4)	0.0172(9)
O(6)	0.5574(8)	0.2572(4)	0.5889(4)	0.0128(9)
O(7)	0.5730(8)	0.0150(4)	0.3892(4)	0.0139(9)
O(8)	0.0757(9)	0.5923(5)	0.2043(4)	0.0217(10)
O(9)	0.2515(10)	0.0390(5)	0.8278(5)	0.0299(13)
O(10)	0.4270(10)	0.4120(5)	0.7963(4)	0.0213(10)
O(11)	0.1250(12)	0.8062(5)	0.8309(5)	0.0312(13)
O(12)	0.2631(11)	0.9566(5)	0.1721(5)	0.0307(13)
O(13)	0.2688(10)	0.6045(4)	0.8040(4)	0.0203(10)
O(14)	0.5703(10)	0.2209(4)	0.7991(5)	0.0225(11)
O(15)	0.3880(10)	0.1887(5)	0.1670(5)	0.0285(12)
X	0	0	0	0.0189(13)
H(1)	0.257(14)	0.423(6)	0.3101(11)	0.023(1)
H(2)	1.046(15)	0.166(6)	0.6838(14)	0.026(1)

*Isotropic for H(1) and H(2).

TABLE 7. SELECTED INTERATOMIC DISTANCES (Å) AND ANGLES (°) FOR TARBAGATAITE

M(1)–O(2)a	2.160(4)		M(2)–O(5)b	2.098(5)	M(3)–O(4)	2.112(4)	
M(1)–O(6)	2.171(4)		M(2)–O(7)	2.098(4)	M(3)–O(1)	2.115(4)	
M(1)–O(5)	2.172(5)		M(2)–O(5)c	2.137(5)	M(3)–O(3)	2.132(4)	
M(1)–O(7)	2.190(4)		M(2)–O(6)	2.207(4)	M(3)–O(6)	2.156(4)	
M(1)–O(3)a	2.192(4)		M(2)–O(7)c	2.233(4)	M(3)–O(4)d	2.179(5)	
M(1)–O(1)	2.226(4)		M(2)–O(2)	2.246(4)	M(3)–O(2)	2.221(4)	
<M(1)–ϕ>	2.185		<M(2)–ϕ>	2.170	<M(3)–ϕ>	2.153	
M(4)–O(4)	2.074(4)	×2	T(1)–O(15)	1.603(5)	T(2)–O(13)d	1.602(4)	
M(4)–O(1)b	2.141(4)	×2	T(1)–O(11)d	1.603(5)	T(2)–O(3)d	1.613(5)	
M(4)–O(3)	2.164(4)	×2	T(1)–O(1)	1.622(4)	T(2)–O(10)d	1.636(5)	
<M(4)–ϕ>	2.126		T(1)–O(13)d	1.644(4)	T(2)–O(8)a	1.638(5)	
			<T(1)–O>	1.618	<T(2)–O>	1.622	
D–O(2)	1.859(5)						
D–O(12)e	1.956(5)		T(3)–O(14)d	1.604(5)	T(4)–O(9)d	1.603(5)	
D–O(9)f	1.957(5)		T(3)–O(6)d	1.611(5)	T(4)–O(12)	1.604(5)	
D–O(11)g	1.961(5)		T(3)–O(8)	1.635(5)	T(4)–O(7)h	1.624(5)	
D–O(15)	1.964(5)		T(3)–O(10)d	1.639(5)	T(4)–O(14)d	1.639(5)	
D–X	2.0450(9)		<T(3)–O>	1.622	<T(4)–O>	1.618	
<D–ϕ>	1.957						
			T(1)d–O(13)–T(2)d	143.1(3)	B–O(15)	2.573(6)	×2
A(1)–O(11)i	2.968(6)		T(3)a–O(14)–T(4)a	143.7(3)	B–O(9)c	2.580(6)	×2
A(1)–O(12)g	2.970(7)		<T–O–T>	143.4	B–O(12)e	2.625(6)	×2
A(1)–O(15)j	2.991(7)				B–O(11)d	2.644(6)	×2
A(1)–O(9)	3.001(7)		A(2)–O(11)i	2.65(5)	B–X	2.6934(1)	×2
A(1)–Xj	3.285(3)		A(2)–O(15)j	2.68(5)	<B–ϕ>	2.593	
A(1)–O(14)	3.367(7)		A(2)–O(12)g	2.76(5)			
A(1)–O(13)k	3.420(7)		A(2)–O(9)	2.80(5)			
A(1)–O(14)b	3.453(7)		A(2)–Xj	2.85(4)			
A(1)–O(13)i	3.480(6)		<A(2)–ϕ>	2.75			
A(1)–O(8)g	3.502(6)						
A(1)–O(10)	3.534(6)		A(1)–A(2)	0.44(5)			
A(1)–O(10)k	3.596(6)						
A(1)–O(8)j	3.687(6)						
<A(1)–ϕ>	3.327						

ϕ = unspecified anion; a: *x*+1, *y*, *z*; b: *x*–1, *y*, *z*; c: –*x*+1, –*y*, –*z*+1; d: –*x*+1, –*y*+1, –*z*+1; e: *x*, *y*–1, *z*; f: –*x*, –*y*, –*z*+1; g: –*x*, –*y*+1, –*z*+1; h: *x*, *y*+1, *z*; i: –*x*, –*y*+1, –*z*+2; j: *x*, *y*, *z*+1; k: –*x*+1, –*y*+1, –*z*+2

unspecified anion). The ideal composition of the *D* site is Ti_2 *apfu*. The four *T* sites are occupied mainly by Si with minor Al (Table 3). The mean T–O distances vary from 1.618 to 1.622 Å, and the T–O–T angles are ~143° (Table 7).

The interstitial A and B sites: In the structure of astrophyllite there are two interstitial sites, the K-dominant *A* site and the Na-dominant *B* site. In tarbagataite, the *A* site is split into two sites, *A*(1) and *A*(2), separated by a short distance (0.44 Å), and which cannot both be occupied at the local scale (*i.e.*, occupancy of one site precludes occupancy of the locally adjacent site). The [13]-coordinated *A*(1) site is occupied by $\square_{1.02}K_{0.76}Rb_{0.18}Cs_{0.04}$ *pfu*, with <A(1)–ϕ> = 3.327 Å; the [5]-coordinated *A*(2) site is occupied by $\square_{1.88}Na_{0.12}$ *pfu*, with <A(2)–ϕ> = 2.75 Å (Tables 7, 8). The occurrence of K and Na at the [13]- and [5]-coordinated *A*(1,2) sites, respectively, has been reported previously for the astrophyllite-group minerals [see details about the splitting of the *A* site by Cámara *et al.* (2010)]. The *A*(1,2) sites give $\square_{0.90}K_{0.76}Rb_{0.18}Na_{0.12}Cs_{0.04}$ *pfu*, and we write the simplified and endmember compositions of the *A* site as $(K,\square)_2$ and $(K\square)$ *pfu*, respectively.

In tarbagataite, the *B* site has a refined site-scattering of 16.58 *epfu*, indicating that a heavier scatterer (rather than Na, 11el) must occur at the *B* site. We assigned $(Ca_{0.62}Na_{0.38})$ *apfu* to the *B* site (calculated site-scattering of 16.58 *epfu*); the ideal composition of the *B* site is Ca *apfu* (Table 8). The *B* site is coordinated by eight O atoms and two X anions, with a <B–ϕ> distance of 2.593 Å. Tarbagataite is the second mineral of the astrophyllite group for which dominance of

TABLE 8. REFINED SITE-SCATTERING AND ASSIGNED SITE-POPULATIONS FOR TARBAGATAITE

Site*	Refined site-scattering (*epfu*)	Assigned site-population (*apfu*)	Calculated site-scattering (*epfu*)	$\langle X\text{–}\phi\rangle_{obs.}$** (Å)	$\langle X\text{–}\phi\rangle_{calc.}$ (Å)
M(1)	48.4(3)	1.40 Mn + 0.55 Fe^{2+} + 0.05 □	49.30	2.185	2.189
M(2)	47.8(3)	1.08 Fe^{2+} + 0.81 Mn + 0.11 □	48.33	2.170	2.168
M(3)	48.5(3)	1.14 Fe^{2+} + 0.73 Mn + 0.08 Mg + 0.05 □	48.85	2.153	2.163
M(4)	23.7(2)	0.74 Fe^{2+} + 0.20 Mg + 0.02 Zr + 0.02 Zn + 0.02 □	23.04	2.126	2.133
D	51.5(3)	1.57Ti + 0.30 Nb + 0.11 Sn + 0.02 □	53.14	1.957	1.991
$^{[13]}$*A*(1)	23.29	1.02 □ + 0.76 K + 0.18 Rb + 0.04 Cs	23.30	3.327	
$^{[5]}$*A*(2)	1.32	1.88 □ + 0.12 Na	1.32	2.75	
$^{[10]}$*B*	16.58	0.62 Ca + 0.38 Na	16.58	2.593	2.626
X		0.65 OH + 0.35 F			
O(4)		2.00 OH			
O(5)		2.00 OH			

*coordination number is shown for a non-octahedral site; X = cation, ϕ = O, OH, F.
**calculated by summing constituent ionic radii; values from Shannon (1976).

TABLE 9. HYDROGEN BONDING IN TARBAGATAITE

D–H…A	D–H (Å)	H…A (Å)	D…A (Å)	∠ DHA (°)
O(4)OH–H(1)…O(11)a	0.98(1)	2.79(6)	3.440(8)	124(5)
O(4)OH–H(1)…O(15)	0.98(1)	2.93(7)	3.537(7)	122(5)
O(5)OH–H(2)…O(9)b	0.98(1)	2.97(7)	3.531(7)	118(5)
O(5)OH–H(2)…O(12)c	0.98(1)	3.00(7)	3.508(8)	113(5)

a: –*x*, –*y*+1, –*z*+1; b: *x*+1, *y*, *z*; c: –*x*+1, –*y*+1, –*z*+1.

TABLE 10. BOND-VALENCES FOR MONOVALENT ANIONS IN TARBAGATAITE

Atom	M(1)	M(2)	M(3)	M(4)	D	A(1)	A(2)	B	H(1)	H(2)	Σ
O(4) OH			0.37 0.32	0.20					0.995		1.89
O(5) OH	0.33	0.40 0.34								0.995	2.07
X**					$0.38^{x2}\rightarrow$	$0.02^{x2}\rightarrow$	$0.01^{x2}\rightarrow$	$0.13^{x2}\rightarrow$			1.08

* bond-valence parameters (*vu*) from Brown (1981) and Brown & Altermatt (1985);
** X = $OH_{0.65}F_{0.35}$;
bond valence incident at the X anion calculated with cation-oxygen and cation-fluorine parameters.

Ca at the *B* site has been recorded. The composition [$Ca(H_2O)$] *pfu* has been reported for the *B* site in sveinbergeite, $Ca(Fe^{2+}{}_6Fe^{3+})Ti_2(Si_4O_{12})_2O_2(OH)_5(H_2O)_4$ (Khomyakov *et al.* 2011).

We write a simplified and endmember $A_2BC_7D_2$ component of the structure for tarbagataite as follows: $(K,\square)_2(Ca,Na)(Fe^{2+},Mn)_7Ti_2$ and $(K\square)CaFe^{2+}{}_7Ti_2$, respectively.

Anion consideration

In tarbagataite, the O(1,3,6-15) atoms belong to (TO_4) tetrahedra and give 24 O atoms *pfu*. Those 24 O atoms correspond to two astrophyllite (Si_4O_{12}) ribbons. There is one site, O(2), which receives bond valence from three M atoms and a D atom, giving O_2 *apfu*. There are two sites, O(4) and O(5), which receive bond valence from three M atoms and are occupied by OH groups. The H(1,2) atoms of the O(4,5) atoms, respectively, are each involved in weak bifurcated hydrogen bonds, with H...A (acceptor) lengths varying from 2.79 to 3.00 Å (Table 9). Bond valence incident at each of the O(4,5) sites sums to 1.89 and 2.07 *vu* (valence unit), respectively (Table 10). The two O(4) and O(5) sites contribute $(OH)_4$ *pfu* to the formula.

The *X* site is occupied by OH groups and F, $OH_{0.65}F_{0.35}$ (Table 8). It receives bond-valences from two D, two A(1), two A(2), and two B atoms, which sum to 1.08 *vu* (Table 10). We were not able to locate an H atom of an OH group at the *X* site.

For tarbagataite, we write the anion part of the structure as $(Si_4O_{12})_2O_2(OH)_4(OH,F)$ (simplified formula) and $(Si_4O_{12})_2O_2(OH)_4(OH)$ *pfu* (endmember formula).

SUMMARY

In accordance with the general formula of the astrophyllite-group minerals $A_2BC_7D_2(T_4O_{12})_2O_2(OH)_4X_{0\text{-}1}$ (Cámara *et al*. 2010, modified after Piilonen *et al*. 2003a), we write the simplified and endmember formulae as $(K,\square)_2(Ca,Na)(Fe^{2+},Mn)_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH,F)$ and $(K\square)CaFe^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH)$, respectively, which agrees with the detailed empirical formula, $(K_{0.76}Rb_{0.18}Na_{0.12}Cs_{0.04}\square_{0.90})_{\Sigma 2}(Ca_{0.62}Na_{0.38})_{\Sigma 1}(Fe^{2+}{}_{3.51}Mn_{2.94}Mg_{0.28}Zr_{0.02}Zn_{0.02}\square_{0.23})_{\Sigma 7}(Ti_{1.57}Nb_{0.30}Sn_{0.11}Zr_{0.02})_{\Sigma 2}(Si_{8.09}Al_{0.03})_{\Sigma 8.12}O_{30.65}H_{4.65}F_{0.35}$, $Z = 1$.

Tarbagataite, ideally $(K\square)CaFe^{2+}{}_7Ti_2(Si_4O_{12})_2O_2(OH)_4(OH)$, is a new member of the astrophyllite group. The crystal structure of tarbagataite is isostructural with those of astrophyllite, niobophyllite, nalivkinite, kupletskite, kupletskite-(Cs), niobokupletskite, and zircophyllite.

Tarbagataite differs from astrophyllite in the composition of the interstitial *A* and *B* sites and the *X* site: $(K,\square)_2$ at the *A* site, (Ca,Na) at the *B* site, and (OH,F) at the *X* site. Tarbagataite and astrophyllite are related by the substitution $^{A}\square + {}^{B}Ca^{2+} + {}^{X}(OH)^- \leftrightarrow {}^{A}K^+ + {}^{B}Na^+ + {}^{X}F^-$.

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