

## THE CRYSTAL CHEMISTRY OF TELYUSHENKOITE AND LEIFITE, $A\ Na_6[Be_2Al_3Si_{15}O_{39}F_2]$ , $A = Cs, Na$

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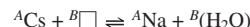
### ABSTRACT

The crystal structures of telyushenkoite, ideally  $Cs\ Na_6[Be_2Al_3Si_{15}O_{39}F_2]$ , trigonal, space group  $P\bar{3}m1$ ,  $a$  14.3770(8),  $c$  4.8786(3) Å,  $V$  873.2(1) Å<sup>3</sup>,  $Z$  = 1, and leifite, ideally  $Na\ Na_6[Be_2Al_3Si_{15}O_{39}F_2]$ , trigonal, space group  $P\bar{3}m1$ ,  $a$  14.361(1),  $c$  4.8570(5) Å,  $V$  867.5(2) Å<sup>3</sup>,  $Z$  = 1, have been refined to  $R$  indices of 2.4(2.9)% based on 874(837) observed ( $4\sigma F$ ) reflections measured with MoK $\alpha$  X-radiation. Electron-microprobe analysis of the crystals used to collect the X-ray intensity data gave the following unit formulae: telyushenkoite: ( $Cs_{0.74}Na_{0.31}K_{0.14}Rb_{0.02}$ )  $Na_6[Be_2Al_{2.06}Zn_{0.30}Si_{15.46}O_{39}F_2]$ ; leifite: ( $Cs_{0.05}Na_{0.57}K_{0.10}Rb_{0.11}$ )  $Na_6[Be_2Al_{1.96}Zn_{0.17}Si_{15.74}O_{39}F_2]$ . There are four tetrahedrally coordinated  $T$  sites with the following site-occupancies:  $T(1) = (Si, Al, Zn)$ ,  $T(2) = T(3) = Si$ ,  $T(4) = Be$ , and the Be coordination is  $O_3F$  with a Be–F distance of 1.576 Å. There is one  $Na$  site occupied by Na and coordinated by seven anions in an augmented trigonal-prismatic arrangement with a  $\langle Na-O_F \rangle$  distance of ~2.545 Å. There is one  $A$  site occupied by large alkali cations with Cs dominant in telyushenkoite and Na dominant in leifite; the  $A$  site is coordinated by six O atoms in telyushenkoite in an octahedral arrangement, and by six O atoms and two ( $H_2O$ ) groups in leifite. The  $B$  site is unoccupied in telyushenkoite and is partly occupied by ( $H_2O$ ) groups in leifite. The  $T(1)$  tetrahedra link to form six-membered rings parallel to {001} that are linked together in both the {001} plane and along [001] by four-membered rings of  $T(2)$  and  $T(3)$  tetrahedra. The six-membered rings of  $T(1)$  tetrahedra stack along the  $c$  direction to form channels that accommodate the  $A$  and  $B$  sites. The  $Na$  polyhedra share edges with the  $T(4)$  (= Be) tetrahedron. The end-member formulae of telyushenkoite and leifite may be written as  $A\ Na_6[Be_2Al_3Si_{15}O_{39}F_2]$ , where  $A = Cs$  and  $Na$ , respectively.

Consideration of the possible end-members of the leifite group led to the following compositions:

	A-site	B-site	Na-site	T-sites and anions
Leifite	Na	( $H_2O$ )	$Na_6$	$[Be_2Al_3Si_{15}O_{39}F_2]$
Telyushenkoite	Cs	□	$Na_6$	$[Be_2Al_3Si_{15}O_{39}F_2]$
–	K	□	$Na_6$	$[Be_2Al_3Si_{15}O_{39}F_2]$
–	Na	( $H_2O$ )	$Na_6$	$[Be_2Zn_{1.5}Si_{16.5}O_{39}F_2]$
–	Cs	□	$Na_6$	$[Be_2Zn_{1.5}Si_{16.5}O_{39}F_2]$
–	K	□	$Na_6$	$[Be_2Zn_{1.5}Si_{16.5}O_{39}F_2]$
–	□	□	$Na_6$	$[Be_2Al_2Si_{16}O_{39}F_2]$
–	□	□	$Na_6$	$[Be_2ZnSi_{17}O_{39}F_2]$

Leifite and telyushenkoite are related by the substitution



Inspection of analytical data in the literature indicate that the K end-member is represented in Nature (although not yet described as a distinct species).

*Keywords:* telyushenkoite, leifite, crystal structure, Cs silicate.

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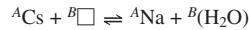
## SOMMAIRE

Nous avons affiné les structures cristallines de la telyushenkoïte, dont la composition idéale est  $\text{Cs Na}_6 [\text{Be}_2 \text{Al}_3 \text{Si}_{15} \text{O}_{39} \text{F}_2]$ , trigonale, groupe spatial  $P\bar{3}m1, a$  14.3770(8),  $c$  4.8786(3) Å,  $V$  873.2(1) Å<sup>3</sup>,  $Z = 1$ , et de la leifite, dont la composition idéale est  $\text{Na Na}_6 [\text{Be}_2 \text{Al}_3 \text{Si}_{15} \text{O}_{39} \text{F}_2]$ , trigonale, groupe spatial  $P\bar{3}m1, a$  14.361(1),  $c$  4.8570(5) Å,  $V$  867.5(2) Å<sup>3</sup>,  $Z = 1$ , jusqu'à un résidu  $R$  de 2.4(2.9)% en utilisant 874(837) réflexions observées ( $4\sigma F$ ) mesurées avec rayonnement MoK $\alpha$ . Une analyse à la microsonde électronique des cristaux utilisés pour le prélèvement des données en diffraction X a mené aux formules suivantes: telyushenkoïte:  $(\text{Cs}_{0.74} \text{Na}_{0.31} \text{K}_{0.14} \text{Rb}_{0.02}) \text{Na}_6 [\text{Be}_2 \text{Al}_{2.06} \text{Zn}_{0.30} \text{Si}_{15.46} \text{O}_{39} \text{F}_2]$ ; leifite:  $(\text{Cs}_{0.50} \text{Na}_{0.57} \text{K}_{0.10} \text{Rb}_{0.11}) \text{Na}_6 [\text{Be}_2 \text{Al}_{1.96} \text{Zn}_{0.17} \text{Si}_{15.74} \text{O}_{39} \text{F}_2]$ . Il y a quatre sites  $T$  à coordinence tétraédrique, remplis selon le schéma suivant:  $T(1) = (\text{Si}, \text{Al}, \text{Zn})$ ,  $T(2) = T(3) = \text{Si}$ ,  $T(4) = \text{Be}$ ; le Be est coordonné par O<sub>3</sub>F, avec une distance Be–F de 1.576 Å. Il y a un site  $Na$ , occupé par le Na et coordonné par sept anions agencés dans un arrangement trigonal prismatique augmenté, avec une distance  $\langle Na-O_F \rangle$  d'environ 2.545 Å. Il y a un site  $A$  site qu'occupe de cations alcalins à large rayon, le Cs étant dominant dans la telyushenkoïte et le Na l'étant dans la leifite; le site  $A$  est entouré de six atomes d'oxygène dans une agencement octaédrique dans la telyushenkoïte, et de six atomes d'oxygène et de deux groupes (H<sub>2</sub>O) dans la leifite. Le site  $B$  est vide dans la telyushenkoïte, et serait partiellement occupé par des groupes (H<sub>2</sub>O) dans la leifite. Les tétraèdres  $T(1)$  sont liés pour former des anneaux à six membres parallèles à {001} qui sont interliés à la fois dans le plan {001} et le long de [001] par des anneaux à quatre membres, tous des tétraèdres  $T(2)$  et  $T(3)$ . Les anneaux à six membres de tétraèdres  $T(1)$  sont empilés le long de la direction  $c$  pour former des canaux qui accommodent les sites  $A$  et  $B$ . Les polyèdres  $Na$  partagent des arêtes avec les tétraèdres  $T(4)$  (= Be). On peut écrire la formule des pôles telyushenkoïte et leifite comme suit:  $A \text{Na}_6 [\text{Be}_2 \text{Al}_3 \text{Si}_{15} \text{O}_{39} \text{F}_2]$ , dans laquelle  $A$  représente Cs et Na, respectivement.

Une considération des formules de pôles possibles dans le groupe de la leifite mène aux compositions suivantes:

	site A	site B	site Na	sites T et anions
Leifite	Na	(H <sub>2</sub> O)	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>3</sub> Si <sub>15</sub> O <sub>39</sub> F <sub>2</sub> ]
Telyushenkoïte	Cs	□	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>3</sub> Si <sub>15</sub> O <sub>39</sub> F <sub>2</sub> ]
–	K	□	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>3</sub> Si <sub>15</sub> O <sub>39</sub> F <sub>2</sub> ]
–	Na	(H <sub>2</sub> O)	Na <sub>6</sub>	[Be <sub>2</sub> Zn <sub>1.5</sub> Si <sub>16.5</sub> O <sub>39</sub> F <sub>2</sub> ]
–	Cs	□	Na <sub>6</sub>	[Be <sub>2</sub> Zn <sub>1.5</sub> Si <sub>16.5</sub> O <sub>39</sub> F <sub>2</sub> ]
–	K	□	Na <sub>6</sub>	[Be <sub>2</sub> Zn <sub>1.5</sub> Si <sub>16.5</sub> O <sub>39</sub> F <sub>2</sub> ]
–	□	□	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>2</sub> Si <sub>16</sub> O <sub>39</sub> F <sub>2</sub> ]
–	□	□	Na <sub>6</sub>	[Be <sub>2</sub> Zn Si <sub>17</sub> O <sub>39</sub> F <sub>2</sub> ]

La leifite et la telyushenkoïte ont une relation exprimée par la substitution



Une inspection des données analytiques déjà dans la littérature montre que le pôle à dominance de K est représenté dans la nature, quoiqu'il n'a pas encore été décrit comme espèce distincte.

(Traduit par la Rédaction)

*Mots-clés:* telyushenkoïte, leifite, structure cristalline, silicate de Cs.

## INTRODUCTION

Telyushenkoïte was described as a new mineral by Agakhanov *et al.* (2001) from the moraine of the Dara-i-Pioz glacier in northern Tajikistan. It is isostructural with leifite, originally described by Bøggild (1915) from Narssârssuk, Greenland. Re-investigation of leifite from this locality (Micheelsen & Petersen 1970) led to the general formula  $[\text{Na}, \{\text{H}_3\text{O}\}]_2 [(\text{Si}, \text{Al}, \text{Be}, \text{B})_7 (\text{O}, \text{F}, \text{OH})_{14}]$ , with  $Z = 3$ . The structure of leifite from Narssârssuk was solved by Coda *et al.* (1974), who revised the formula to  $\text{Na}_6 [\text{Si}_{16} \text{Al}_2 (\text{Be} X)_2 \text{O}_{39}] \bullet 1.5\text{H}_2\text{O}$ , where  $X = (\text{OH}, \text{F})$ . The chemical composition of Micheelsen & Petersen (1970) does not correspond very well to the structure. The discovery of telyushenkoïte, a new Cs-bearing mineral with a strong structural affinity to leifite, seemed the ideal opportunity to characterize

role of Cs in this structure type and to resolve any ambiguities in the structure and chemical composition of leifite.

## EXPERIMENTAL

The telyushenkoïte crystals used in this work are equant transparent-to-white crystals from the type locality. The leifite crystals are transparent-to-white hexagonal prisms with striations parallel to the  $c$  axis, and are from Mont Saint-Hilaire, Quebec, Canada. For each sample, a small crystal was attached to a glass fiber and mounted on a Siemens P4 automated four-circle diffractometer equipped with MoK $\alpha$  X-radiation. Reflections over the range  $4^\circ \leq 2\theta \leq 25^\circ$  were centered, and the unit-cell dimensions (Table 1) were refined by least-squares from the resultant setting angles. Intensity

data were collected in  $\theta$ -2 $\theta$  scan-mode at a fixed scan-rate of 2.0°/20/min. A total of 1896 (1882) reflections for telyushenkoite (leifite) was measured over the range  $4^\circ \leq 2\theta \leq 60^\circ$ . Two standard reflections were monitored every fifty-eight reflections; there were no significant changes in their intensities during data collection. Psi-scan data were measured on 20 reflections out to  $60^\circ/2\theta$  at increments of  $5^\circ$ , and an absorption correction, modeling the crystal as a triaxial ellipsoid, reduced  $R$ (azimuthal) from 1.8(2.1) to 0.9(1.1)%. The data were corrected for Lorentz, polarization and background effects, averaged and reduced to structure factors; of the 953(946) unique reflections, 874(837) were considered as observed [ $|F_o| \geq 4\sigma F$ ].

#### ELECTRON-MICROPROBE ANALYSIS

The crystals used in the collection of the X-ray intensity data, together with two other samples of leifite (MSH1 and MSH2) from Mont Saint-Hilaire, Quebec, Canada, and one sample (KAR) from Mt. Karnašurt, Kola Peninsula, Russia, were analyzed with a Cameca SX-50 electron microprobe operating in wavelength-dispersion mode with an accelerating voltage of 15 kV, a specimen current of 20 nA, a beam size of 20  $\mu\text{m}$  and counting times on peak and background of 2 and 10 s, respectively. The following standards and crystals were used for  $K$  or  $L$  X-ray lines: F: fluorine-dominant analogue of riebeckite, TAP; Na, Al: albite, TAP; Si: albite, PET; K: orthoclase, PET; Ca: diopside, PET; Zn: gahnite, LiF; Rb: Rb-bearing microcline, TAP; Cs: pollucite, PET. Data were reduced using the  $\phi(\rho Z)$  procedure of Pouchou & Pichoir (1985). The chemical com-

position of the fragment used in the collection of the X-ray intensity data is given in Table 2 and is the mean of 25 determinations. The unit formula was calculated on the basis of forty oxygen atoms (*i.e.*, excluding F), assuming  $\text{Be} = 2 \text{ apfu}$ . For telyushenkoite, an analytical value for  $\text{BeO}$  (3.53 wt.% BeO) was presented by Agakhanov *et al.* (2001); it compares well with the calculated value of 3.46 wt.% BeO.

#### STRUCTURE REFINEMENT

Scattering curves for neutral atoms were taken from the International Tables for Crystallography (1992).  $R$  indices are of the form given in Table 1, and are expressed as percentages. The Siemens SHELXTL PLUS (PC version) system of programs was used for this work.

Using the atom coordinates of Coda *et al.* (1974), the structures refined rapidly to an  $R$  index of ~3% for a model with variable scattering at the cation sites having coordination numbers between [6] and [8], and anisotropic displacements for all atoms. For telyushenkoite, the high refined scattering value at the *A site* indicates that Cs occupies this site; the scattering curve was thus assigned and the site occupancy refined. For leifite, chemical analysis showed the unit formula to contain K and Na in excess of that needed to fill the *Na* site, and the occupancies of the *A* and *B* sites were refined with

TABLE 2. CHEMICAL COMPOSITION (wt.%) and UNIT FORMULAE (apfu) FOR TELYUSHENKOITE (T) AND LEIFITE (L)

	T	L	MSH1***	MSH2***	KAR**
$\text{SiO}_2$	64.32	68.86	69.96	68.32	69.65
$\text{Al}_2\text{O}_3$	7.26	7.29	8.50	9.69	8.05
$\text{ZnO}$	1.71	1.02	0.09	0.37	0.71
$\text{BeO}^*$	3.46**	3.64	3.72	3.71	3.71
$\text{Na}_2\text{O}$	13.53	14.82	14.75	14.17	14.78
$\text{K}_2\text{O}$	0.47	0.35	0.51	2.00	0.71
$\text{Cs}_2\text{O}$	7.25	0.48	0.09	0.01	0.04
$\text{Rb}_2\text{O}$	0.15	0.72	0.42	0.51	0.31
F	2.84	2.34	2.93	2.86	2.99
O=F	-1.20	-0.99	-1.23	-1.20	-1.26
Total	99.79	98.54	99.74	100.43	99.69
Si	15.49	15.74	15.66	15.34	15.67
Al	2.06	1.96	2.24	2.56	2.13
Zn	0.30	0.17	0.02	0.06	0.12
Be	2.00	2.00	2.00	2.00	2.00
$\Sigma T$	19.85	19.87	19.92	19.96	19.92
Na	6.00	6.00	6.00	6.00	6.00
K	0.14	0.10	0.15	0.57	0.20
Na	0.31	0.57	0.39	0.17	0.44
Cs	0.74	0.05	0.01	0.00	0.00
Rb	0.02	0.11	0.06	0.07	0.05
$\Sigma$	1.21	0.83	0.61	0.81	0.69
F	2.16	1.69	2.07	2.03	2.12

\* calculated for  $\text{Be} = 2 \text{ apfu}$

\*\* The value determined colorimetrically for  $\text{BeO}$  for a bulk sample of telyushenkoite is 3.53 wt.%

\*\*\* MSH = Mont Saint-Hilaire, Quebec, Canada;

KAR = Mt. Karnašurt, Russia

TABLE 1. MISCELLANEOUS REFINEMENT DATA FOR TELYUSHENKOITE AND LEIFITE

	telyushenkoite	leifite
a (Å)	14.3770(8)	14.3608(11)
c	4.8786(3)	4.8570(5)
$V (\text{\AA}^3)$	873.2(1)	867.5(2)
Space group	$P\bar{3}m1$	$P\bar{3}m1$
Z	1	1
$D_{\text{calc}}$ (g/cm <sup>3</sup> )	2.734	2.624
Absorption coefficient (mm <sup>-1</sup> )	1.83	1.15
$F(000)$	702.9	678.1
Crystal size (mm)	0.08 x 0.16 x 0.17	0.10 sphere
Radiation	MoK $\alpha$	MoK $\alpha$
2 $\theta$ -range for data collection (°)	4–60	4–60
$R(\text{int})$ (%)	1.6	1.9
Total no. of reflections	1896	1882
Independent reflections	953	946
$F_o > 4\sigma(F)$	874	837
Refinement method	Full-matrix least-squares on $F^2$ ; fixed weights proportional to $1/\sigma(F^2)$	
Goodness of fit on $F^2$	1.097	1.062
Final $R$ indices [ $F_o > 4\sigma(F)$ ] (%)	2.4	2.9
$R$ indices (all data) (%)	2.7	3.3
$wR_2$ (%)	7.4	9.1
Goof	1.097	1.062

TABLE 3. FINAL ATOMIC PARAMETERS FOR TELYUSHENKOITE (upper row) AND LEIFITE (lower row)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>23</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>12</sub>
<i>T</i> (1)	0	0.21677(5)	1/2	0.0129(2)	0.0137(3)	0.0143(2)	0.0105(3)	-0.0007(1)	-0.0015(2)	0.0068(2)
	0	0.21681(5)	1/2	0.0137(2)	0.0141(4)	0.0145(3)	0.0123(4)	-0.0010(1)	-0.0020(2)	0.0070(2)
<i>T</i> (2)	0	0.34441(5)	0	0.0105(2)	0.0117(3)	0.0106(2)	0.0096(3)	-0.0002(1)	-0.0004(2)	0.0058(2)
	0	0.34443(5)	0	0.0113(2)	0.0120(3)	0.0113(3)	0.0108(3)	-0.0002(1)	-0.0003(2)	0.0060(2)
<i>T</i> (3)	0.44759(2)	-0.44759(2)	0.3068(1)	0.0078(2)	0.0092(2)	0.0092(2)	0.0074(3)	0.0002(1)	-0.0002(1)	0.0063(2)
	0.44762(3)	-0.44762(3)	0.3059(1)	0.0075(2)	0.0093(3)	0.0093(3)	0.0062(3)	0.0001(1)	-0.0001(1)	0.0062(3)
<i>T</i> (4)	1/3	2/3	0.367(1)	0.0083(8)	0.0082(13)	0.0082(13)	0.0086(20)	0	0	0.0041(6)
	1/3	2/3	0.366(1)	0.0091(9)	0.0091(14)	0.0091(14)	0.0092(22)	0	0	0.0045(7)
Na	0.75039(5)	-0.75039(5)	0.2029(2)	0.0212(3)	0.0213(4)	0.0213(4)	0.0161(5)	-0.0033(2)	0.0033(2)	0.0072(5)
	0.75042(5)	-0.75042(5)	0.2033(3)	0.0214(3)	0.0212(5)	0.0212(5)	0.0165(6)	-0.0031(2)	0.0030(2)	0.0066(5)
A	0	0	0	0.0275(2)	0.0312(3)	0.0312(3)	0.0200(3)	0	0	0.0156(1)
	0	0	0	0.0444(11)	0.0495(14)	0.0495(14)	0.0343(17)	0	0	0.0247(7)
B	-	-	-	-	-	-	-	-	-	-
	0	0	1/2	0.0297(40)	0.0119(28)	0.0119(28)	0.0653(75)	0	0	0.0060(14)
O(1)	0.1009(1)	-0.1009(1)	0.3920(5)	0.0340(6)	0.0349(9)	0.0349(9)	0.0200(10)	0.0067(5)	-0.0067(5)	0.0084(11)
	0.1008(1)	-0.1008(1)	0.3946(5)	0.0325(6)	0.0328(9)	0.0328(9)	0.0190(11)	0.0063(5)	-0.0063(5)	0.0067(11)
O(2)	0.3081(1)	0.2612(1)	0.2473(3)	0.0193(3)	0.0229(7)	0.0160(6)	0.0187(6)	0.0075(5)	0.0089(5)	0.0097(5)
	0.3078(1)	0.2614(1)	0.2486(3)	0.0178(3)	0.0201(7)	0.0156(7)	0.0181(7)	0.0068(5)	0.0080(5)	0.0094(6)
O(3)	0.3597(1)	0.4585(1)	0.1047(2)	0.0127(3)	0.0144(6)	0.0112(6)	0.0135(5)	-0.0030(4)	-0.0038(4)	0.0072(5)
	0.3595(1)	0.4585(1)	0.1035(3)	0.0124(3)	0.0138(6)	0.0111(6)	0.0132(6)	-0.0033(4)	-0.0042(5)	0.0069(5)
O(4)	1/2	0	1/2	0.0145(5)	0.0186(9)	0.0082(11)	0.0132(11)	-0.0038(9)	-0.0019(4)	0.0041(5)
	1/2	0	1/2	0.0133(5)	0.0165(9)	0.0092(11)	0.0116(11)	-0.0034(9)	-0.0017(4)	0.0046(6)
O(5)	0.39440(7)	-0.39440(7)	0.4825(3)	0.0107(3)	0.0141(6)	0.0141(6)	0.0085(7)	-0.0006(3)	0.0006(3)	0.0106(7)
	0.39431(7)	-0.39431(7)	0.4821(3)	0.0105(4)	0.0137(6)	0.0137(6)	0.0084(8)	-0.0005(3)	0.0006(3)	0.0099(7)
F	1/3	2/3	0.0438(5)	0.0136(5)	0.0175(8)	0.0175(8)	0.0058(10)	0	0	0.0088(4)
	1/3	2/3	0.0421(5)	0.0136(5)	0.0167(8)	0.0167(8)	0.0073(11)	0	0	0.0084(4)

TABLE 4. SELECTED INTERATOMIC DISTANCES (Å) AND ANGLES (°) FOR TELYUSHENKOITE AND LEIFITE

	telyushenkoite	leifite	telyushenkoite	leifite	
T(1)–O(1)	x2 1.656(1)	1.650(1)	T(2)–O(2)	x2 1.592(1)	1.589(1)
T(1)–O(2)	x2 1.678(1)	1.665(1)	T(2)–O(3)	x2 1.625(1)	1.622(1)
<T(1)–O>	1.667	1.658	<T(2)–O>	1.609	1.606
T(3)–O(3)	x2 1.639(1)	1.637(1)	T(4)–O(5)	x3 1.622(2)	1.618(3)
T(3)–O(4)	1.610(1)	1.608(1)	T(4)–F	1.576(6)	1.575(6)
T(3)–O(5)	1.578(2)	1.578(2)	<T(4)–(O,F)>	1.611	1.607
<T(3)–O>	1.617	1.615			
Na–O(2)	x2 2.842(2)	2.845(2)	A–O(1)	x6 3.158(3)	3.157(3)
Na–O(3)	x2 2.454(1)	2.445(1)	A–B	x2 2.439(1)	2.429(1)
Na–O(5)	x2 2.418(1)	2.411(1)			
Na–F	2.407(2)	2.400(2)			
<Na–O,F>	2.548	2.543			
T(1)–O(1)–T(1)	140.5(1)	141.4(2)			
T(1)–O(2)–T(2)	137.96(9)	138.32(9)			
T(2)–O(3)–T(3)	132.83(9)	132.60(9)			
T(3)–O(4)–T(3)	180.00	180.00			
T(3)–O(5)–T(4)	126.7(2)	126.8(2)			
<T–O–T>	145.6	145.8			

K and O ( $\equiv H_2O$ ), respectively, as the variable scattering species. Refinement converged rapidly to  $R$  indices of 2.4 and 2.9%, respectively, for telyushenkoite and leifite. Positional and displacement parameters for both structures are given in Table 3, selected interatomic distances in Table 4, site-scattering parameters and assigned site-populations in Table 5, and bond-valences in Table 6. Observed and calculated structure-factors are available from The Depository of Unpublished Data, CISTI, National Research Council, Ottawa, Ontario K1A 0S2, Canada.

#### DESCRIPTION OF THE STRUCTURE

##### *Coordination of the cations*

There are four  $T$  sites in this structure, each surrounded by four anion sites in a tetrahedral arrangement. The site-scattering values from these sites indicate that the  $T(1)$ ,  $T(2)$  and  $T(3)$  sites are occupied by atoms with atomic numbers close to 14, whereas the  $T(4)$  site is occupied by an atom(s) with an atomic number close to 4. The unit formulae for these crystals indicate that  $T(1)$ ,  $T(2)$  and  $T(3)$  are occupied by Si and Al, and  $T(4)$  is occupied by Be. In both telyushenkoite and leifite,  $\langle T(1)-O \rangle \approx 1.663 \text{ \AA}$  and  $\langle T(2)-O \rangle \approx \langle T(3)-O \rangle \approx 1.612 \text{ \AA}$ , indicating that Al must be highly to completely ordered at the  $T(1)$  site. The  $\langle T(4)-O, F \rangle$  distance is  $\sim 1.609 \text{ \AA}$ , significantly shorter than the grand  $\langle Be-\phi \rangle$  distance of  $1.633 \text{ \AA}$  for all minerals given by Hawthorne & Huminicki (2002). However, this is the result of one of the anions of the tetrahedron being F rather than O. The mean anion-coordination number for the anions coordinating the  $T(4)$  cation is [4] (Table 6). The relevant anion radii (Shannon 1976) are  $1.38 \text{ \AA}$  for  $^{[4]}O$ ,

$1.30 \text{ \AA}$  for  $^{[4]}F$ . The  $T(4)$  tetrahedron involves one F anion. Hence the relative shortening of the bond due to the presence of one F in the tetrahedron is  $(1.38 - 1.30)/4 = 0.02 \text{ \AA}$ . Correcting the  $\langle T(4)-O, F \rangle$  distance by this amount gives  $1.629 \text{ \AA}$ , close to the grand  $\langle Be-\phi \rangle$  distance of  $1.633 \text{ \AA}$ . Thus the size of the  $T(4)$  tetrahedron is in accord with complete  $T(4)$  occupancy by Be.

The  $Na$  site is occupied solely by Na in both leifite and telyushenkoite, and is surrounded by six O atoms and one F atom in an augmented trigonal prismatic arrangement, with  $\langle Na-O, F \rangle$  distances of  $\sim 2.545 \text{ \AA}$ , in fair accord with the sum of the constituent ionic radii (Shannon 1976):  $1.35 + 1.12 = 2.47 \text{ \AA}$ . The A site is surrounded by six anions in an octahedral arrangement, with a  $\langle A-O \rangle$  distance of  $\sim 3.158 \text{ \AA}$ . In telyushenkoite, the refined site-scattering value at the A site indicates occupancy by a heavy scatterer, in accord with the unit formula derived from the chemical composition (Table 2). Assigning the larger and heavier scattering species to A results in the same site-scattering value as was obtained from the refinement. In leifite, the refined site-scattering value at A is much lower than that for telyushenkite (Table 5), and is reasonably well in accord with the large-cation content obtained by electron-microprobe analysis (Table 5). In telyushenkoite, the B site is not occupied. When the site scattering at this site was refined with both Na and O as the scattering species, the occupancy refined to zero (within one standard deviation) for both scattering species. In leifite, the same procedure resulted in significant occupancy of the B site (Table 5). The total refined scattering at the A and B sites in leifite is  $18.7 \text{ epfu}$  (electrons per formula unit), significantly larger than the value of  $15.0 \text{ epfu}$

TABLE 5. SITE-SCATTERING FACTORS (RSSF; epfu), ASSIGNED SITE POPULATIONS (apfu) AND CALCULATED SITE-SCATTERING FACTORS (CSSF; epfu).

Site	RSSF	Site population	CSSF
<i>Telyushenkoite</i>			
A	45.3(2)	$0.74 \text{ Cs} + 0.02 \text{ Rb} + 0.14 \text{ K} + 0.10 \text{ Na}$	45.2
B	—	—	
Na*	(66)	6 Na	66
T(1)	(86.7)*	$3.64 \text{ Si} + 2.06 \text{ Al} + 0.30 \text{ Zn}$	86.7
T(2)	(84)*	6 Si	84
T(3)	(84)*	6 Si	84
T(4)	(8)*	2 Be	84
<i>Leifite</i>			
A	13.7(2)	$0.05 \text{ Cs} + 0.11 \text{ Rb} + 0.10 \text{ K} + 0.57 \text{ Na}$	15.0
B	5.0(2)	$0.63 \text{ (H}_2\text{O)}$	5.0
Na*	(66)	6 Na	66
T(1)	84.9(4)	$3.87 \text{ Si} + 1.96 \text{ Al} + 0.17 \text{ Zn}$	84.8
T(2)	(84)*	6 Si	84
T(3)	(84)*	6 Si	84
T(4)	(8)*	2 Be	84

\* values in parentheses were not refined

TABLE 6. BOND-VALENCE TABLES

telyushenkoite *						
T(1)	T(2)	T(3)	T(4)	Na	A	$\Sigma$
O(1)	$0.95^{+2} \text{ I}^-$				$0.12^{+6} \text{ I}$	2.02
O(2)	$0.90^{+2} \text{ I}$	$1.01^{+2} \text{ I}$		$0.10^{+2} \text{ I}$		2.11
O(3)		$1.01^{+2} \text{ I}$	$0.98^{+2} \text{ I}$	$0.19^{+2} \text{ I}$		2.18
O(4)			$1.05^{+2} \text{ I}^-$			2.10
O(5)			1.15	$0.51^{+3} \text{ I}$	$0.19^{+2} \text{ I}^-$	2.04
F				0.46	$0.14^{+3} \text{ I}^-$	0.88
$\Sigma$	3.70	4.04	4.16	1.99	1.10	0.72
leifite						
T(1)	T(2)	T(3)	T(4)	Na	$^a\text{Na}^{**}$	$\Sigma$
O(1)	$0.95^{+2} \text{ I}^-$				$0.06^{+6} \text{ I}$	1.93
O(2)	$0.92^{+2} \text{ I}$	$1.12^{+2} \text{ I}$		$0.10^{+2} \text{ I}$		2.14
O(3)		$1.02^{+2} \text{ I}$	$0.98^{+2} \text{ I}$	$0.19^{+2} \text{ I}$		2.19
O(4)			$1.06^{+2} \text{ I}^-$			2.12
O(5)			1.15	$0.51^{+3} \text{ I}$	$0.20^{+2} \text{ I}^-$	2.06
F				0.46	$0.15^{+3} \text{ I}^-$	0.91
$^a\text{H}_2\text{O}$					$0.18^{+2} \text{ I}^-$	0.36
$\Sigma$	3.74	4.28	4.17	1.99	1.13	0.76

\* after Brown & Altermatt (1985);

\*\*calculated for Na at A bonded to  $(\text{H}_2\text{O})$  at B.

available from the cations in the unit formula (Table 2). Thus there is an “invisible” component at the *B* site in leifite. As this site is only  $\sim 2.44 \text{ \AA}$  from the *A* site, it must bond to the *A*-site cations (as the amount of vacancy at *A*, 0.17  $\square \text{ pfu}$  is insufficient to “coordinate” the species at the *B* site). Thus ( $\text{H}_2\text{O}$ ) was assigned to the *B* site. The amount of ( $\text{H}_2\text{O}$ ) at *B* is approximately the same as the amount of Na at *A*, suggesting that  ${}^4\text{Na}$  bonds to  ${}^8\text{H}_2\text{O}$ . Inspection of Table 6 shows that this must be the case. If the *B* site were vacant where adjacent *A* sites were occupied by Na, then the incident bond-valence at  ${}^4\text{Na}$  would be only 0.36 *vu*. Where  ${}^4\text{Na}$  is bonded to ( $\text{H}_2\text{O}$ ) at *B*, the incident bond-valence at  ${}^4\text{Na}$  is 0.76 *vu*, similar to that in telyushenkoite (Table 6). So what happens in telyushenkoite where the *A* site is occupied by a small amount (0.10 *apfu*) of Na? The site-scattering value at *B* that would result from coordination of this amount of Na at *A* is  $\sim 0.8 \text{ epfu}$ , not significantly different (*i.e.*,  $<$  three standard deviations) from zero, as was observed in the refinement. Thus the *B* site could possibly be occupied by sufficient ( $\text{H}_2\text{O}$ ) to satisfy the bond-valence requirements of Na at the *A* site in telyushenkoite.

### Structure topology

Projected down the *c* axis (Fig. 1), the *T*(1) tetrahedra form six-membered rings of vertex-sharing (Si,Al)-bearing tetrahedra that are linked together by four-membered rings of *T*(2) and *T*(3) tetrahedra. Triplets of adjacent four-membered *T*(2)–*T*(3) rings are linked by a *T*(4) (=Be) tetrahedron to form seven-membered rings involving all four tetrahedra. The four-membered *T*(2)–*T*(3) rings also link the six-membered *T*(1) rings in the *c* direction (Fig. 2), and themselves link in the same direction by sharing vertices of *T*(3) tetrahedra *via* a straight (=  $180^\circ$ ) *T*–O–*T* linkage [*T*(3)–O–*T*(3), Table 4]. The six-membered rings of *T*(1) tetrahedra stack along [001] (Fig. 2) to form channels parallel to the *c* axis, and the *A* and *B* sites occur within these channels. The Na site occurs within the channels formed by the seven-membered rings involving all four tetrahedra (Fig. 1). Three Na polyhedra share edges with the *T*(4) tetrahedron (Fig. 3a), and these  $[\text{Na}_3(\text{Be O}_4)\text{O}_{13}\text{F}]$  clusters share vertices along the *c* direction (Fig. 3b). As a result, the *F* site is tetrahedrally coordinated by one *B* and three Na atoms; hence there can be no substi-

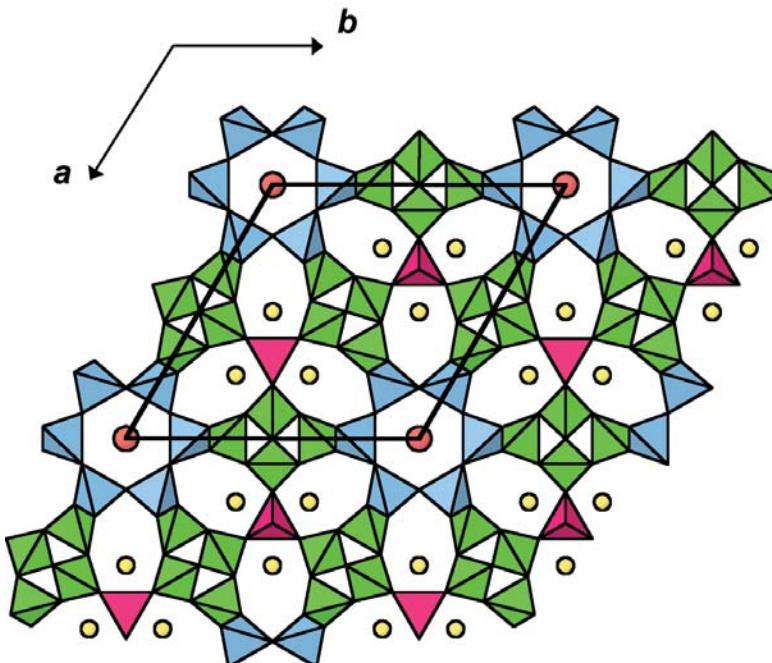


FIG. 1. The crystal structure of telyushenkoite viewed down [001]; the *A* and *Na* sites are shown as red and yellow circles, respectively, *T*(1) tetrahedra [= Si,Al] are blue, *T*(2) and *T*(3) tetrahedra [= Si] are green, and *T*(4) [= Be] tetrahedra are magenta.

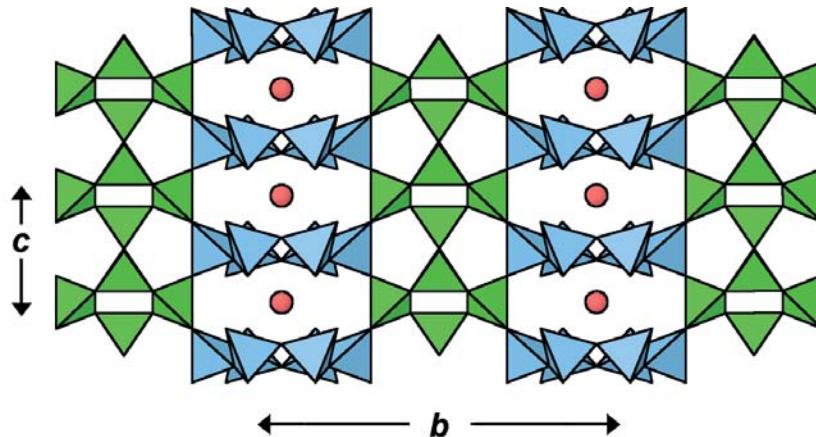


FIG. 2. A fragment of the crystal structure of telyushenkoite projected onto (100), showing the linkage between the six-membered rings of  $T(1)$  tetrahedra and the four-membered rings of  $T(2)$  and  $T(3)$  tetrahedra; legend as in Figure 1.

tution of (OH) for F at this site as there is no room for the H atom within the tetrahedron of cations surrounding the F site.

#### *Chemical compositions*

The end-member formulae for these two minerals have  $A = \text{Cs}$  or  $\text{Na}$ ,  $B = \square$  or  $\text{H}_2\text{O}$ , and  $Na = \text{Na}$ . As the anion charge is  $39 \times 2 + 2 = 80^-$ , the total charge of the tetrahedrally coordinated cations must be  $80 - 7 = 73^+$ . The results of the crystal-structure refinement show that  $T(2) = T(3) = \text{Si}$  and  $T(4) = \text{Be}$ , a total charge of  $12 \times 4 + 2 \times 2 = 52^+$ . This means that the aggregate charge at the  $T(1)$  site is  $73 - 52 = 21^+$ . The  $T(1)$  site populations indicated from the unit formulae involve Si, Al and Zn. The possible end-member compositions resulting in the required total charge at the  $T(1)$  site are  $(\text{Si}_3 \text{Al}_3)$  and  $(\text{Si}_{4.5} \text{Zn}_{1.5})$ . Both telyushenkoite and leifite are closer to  $T(1) = (\text{Si}_3 \text{Al}_3)$ , and hence the end-member compositions for these two minerals are  $A \text{Na}_6 [\text{Be}_2 \text{Al}_3 \text{Si}_{15} \text{O}_{39} \text{F}_2]$  with  $A = \text{Cs}$  and  $\text{Na}$ , respectively.

Petersen *et al.* (1994), Men'shikov *et al.* (1999) and Yakovenchuk *et al.* (1999) reported chemical compositions of leifite from Ilímaussaq, Greenland, Vester Arøya, Norway, Lovozero, Russia, and Khibina, Russia, that are close to the end-member  $T$ -site composition ( $\text{Be}_2 \text{Al}_3 \text{Si}_{15}$ ). However, most leifite compositions (Petersen *et al.* 1994, Larsen & Åsheim 1995, this study) have  $T\text{Al} \approx 2 \text{ apfu}$  (atoms per formula unit) and obviously depart significantly from the ideal end-member composition. Figure 4a shows the variation in  $^{[4]}\text{Al}$  as a function of Si. There is a cluster of points around the ideal end-member composition  $\text{Na Na}_6 [\text{Be}_2 \text{Al}_3 \text{Si}_{15} \text{O}_{39} \text{F}_2]$ , and the rest of the data trend away from the point

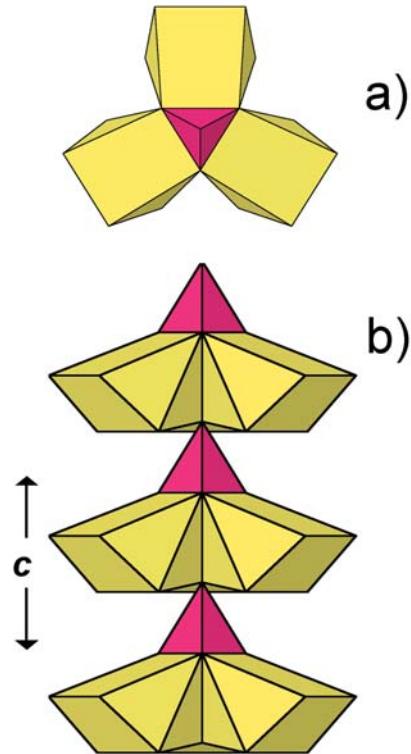


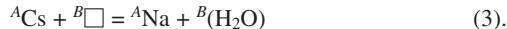
FIG. 3. A detail of the crystal structure of telyushenkoite: (a) a cluster of one  $T(4)$  tetrahedron and three [7]-coordinated  $\text{Na}$  polyhedra projected onto (001); (b) linkage of these clusters along [001]. The  $T(4)$  tetrahedra are magenta, and the  $\text{Na}$  polyhedra are yellow.

with decreasing  $[{}^4\text{Al}]$  and increasing Si. Two lines are shown on Figure 4, representing the substitutions



It is apparent that most of the data departing from the end-member composition do so in a direction intermediate to these two substitutions. Figure 4b shows the variation in A-site occupancy as a function of Si content. As in Figure 4a, telyushenkoite lies on the line corresponding to substitution (1), and the other data points lie between (or close to) the lines corresponding to substitutions (1) and (2). The data from Petersen *et al.* (1994) actually fall slightly outside this range; however, they did not detect any Rb in their samples, which seems unusual as we detected significant Rb in telyushenkoite and in leifite from Mont Saint-Hilaire.

Let us now consider possible variation in cation occupancy at the A site. Leifite from Narssârssuk, Greenland, was originally analyzed by Bøggild (1915), who did not report any K present. In their re-analysis of the type material, Micheelsen & Petersen (1970) did not detect any K or Zn with the electron microprobe, indicating Na to be the dominant species at the A site. Telyushenkoite has Cs dominant at A, and hence is the Cs analogue of leifite. However, previous analyses of leifite have reported  $\text{H}_2\text{O}$  in quantities up to ~1 wt%, and in our structure refinement of leifite from Mont Saint-Hilaire, we assign 0.63 ( $\text{H}_2\text{O}$ ) groups *pfu* to the B site and suggest that it bonds to Na at the A site. Conversely, in telyushenkoite, there is no detectable ( $\text{H}_2\text{O}$ ) at the B site, suggesting that leifite and telyushenkoite are related by the substitution



Inspection of chemical compositions of leifite *sensu lato* (e.g., MSH2, Table 2, this study; Petersen *et al.* 1994, Larsen & Åsheim 1995) show samples with K dominant at the A site; these samples are the K equivalent of leifite and telyushenkoite, and warrant species status.

An area of uncertainty in the crystal chemistry of the leifite-group minerals is the role of H in the structure. Most chemical compositions of leifite were determined by electron-microprobe analysis and hence lack ( $\text{H}_2\text{O}$ ) determinations. However, chemical analysis of some leifite samples (e.g., Micheelsen & Petersen 1970, Larsen & Åsheim 1995) show the presence of H (given as  $\text{H}_2\text{O}$  in the chemical compositions). Larsen & Åsheim (1995) give spectroscopic evidence that H is present in the structure of leifite from Langesundsfjord, Norway, as ( $\text{OH}$ ), whereas other investigators have assumed that H occurs in leifite as ( $\text{H}_2\text{O}$ ). In leifite from Mont Saint-Hilaire, Quebec, examined here, ( $\text{H}_2\text{O}$ ) was assigned to the B site, and an argument is made that the electron density observed at the B site in the crystal-structure refinement *must* be due to ( $\text{H}_2\text{O}$ ); there is no other scattering species that could possibly occupy this site. Can H occur as ( $\text{OH}$ ) in the structure of leifite? If it does, then we are left with the question of where to put it in the structure. Hydroxyl cannot occupy the B site, as there is insufficient bond-valence incident at the site to accommodate ( $\text{OH}$ ). Thus the only other alternative is that ( $\text{OH}$ ) substitutes for another anion in the structure. There are two possibilities here: (1)  $(\text{OH})^- \rightleftharpoons \text{F}^-$  substitution at the F site; (2)  $(\text{OH})^- \rightleftharpoons \text{O}^{2-}$  substitution at any O site in the structure. (1) Above, we make the argument that ( $\text{OH}$ ) cannot substitute for F at the F site because of stereochemical constraints. This argument is

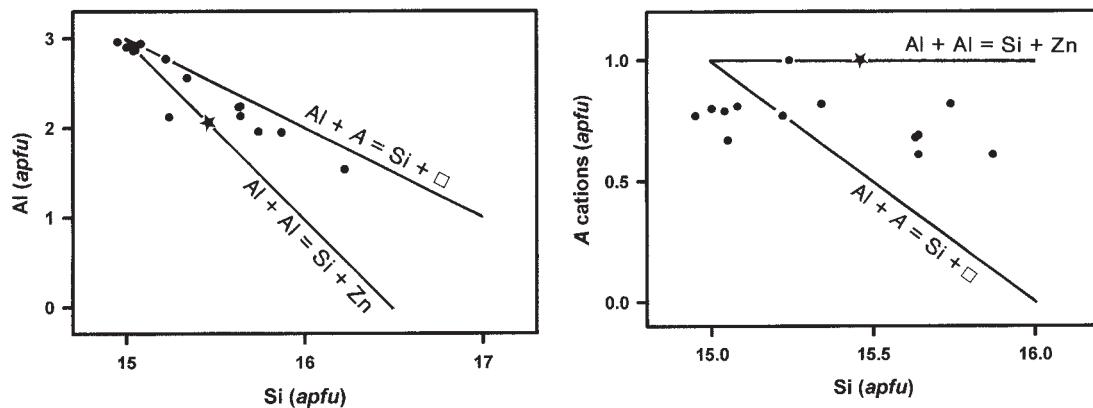


FIG. 4. Chemical relations in leifite *sensu lato* and telyushenkoite; (a) Al as a function of Si; (b) A cations as a function of Si. Substitutions (1) and (2) [see text] are shown, and the star indicates telyushenkoite. The compositions are taken from Petersen *et al.* (1994), Khomyakov *et al.* (1979), Men'shikov *et al.* (1999) and Yakovenchuk *et al.* (1999).

in accord with the observation that  $F \approx 2 \text{ apfu}$  in most leifite samples, including the leifite of Larsen & Åsheim (1995), in which they have assigned ~1 atom of H *pfa*. (2) The only possibility for (OH) to substitute for  $O^{2-}$  is at the O(1) site. This site links to two  $T(1)$  cations (Table 6), and hence substitution of Be for Si at adjacent  $T(1)$  sites would give an incident bond-valence of ~1 *vu* (valence units) at the linking O(1) anion. This would provide a problem at the adjacent O(1) sites that link to Be and Si for an incident bond-valence sum of ~1.5 *vu*. However, if the Be occupying  $T(1)$  always completely filled the six tetrahedra of the six-membered ring of  $T(1)$  tetrahedra (Fig. 1) at the local scale, then six (OH) groups would each be coordinated by two Be atoms in the ring. This produces a net charge-deficiency within the structure, *i.e.*,  ${}^{T(1)}\text{Be} + {}^{O(1)}(\text{OH}) \rightleftharpoons {}^{T(1)}\text{Si} + {}^{O(1)}\text{O}^{2-}$ , and this can only be compensated for by substitution of Si for Al to give a net composition of  ${}^4\text{Na} {}^{Na_6} [\text{Be}_2 (\text{Al}_2\text{Si}) (\text{BeSi}_{14}) \text{O}_{38} (\text{OH}) \text{F}_2]$ ; the resulting composition of the framework is  $[\text{Be}_3 \text{Al}_2 \text{Si}_{15} \text{O}_{38} (\text{OH}) \text{F}_2]$ . This seems to be the only way in which (OH) could be incorporated into the structure of leifite. However, the composition of the leifite sample of Larsen & Åsheim (1995) is  $[\text{Be}_{2.07} \text{Al}_{3.04} \text{Si}_{14.66}]$ , which does not correspond in any way with the composition derived above for an (OH)-bearing leifite. A more extensive infrared spectroscopic study of leifite is needed to resolve this issue directly.

#### Possible end-member compositions

In the discussion on chemical composition given above, several end-member compositions were identified within the leifite group; these are listed in Table 7. Inspection of Table 2 shows that the occupancy of the A site may be less than 1.0 *apfu*, giving rise to end-member compositions with the A-site vacant. The corresponding Zn-free and Al-free end-member compositions are listed in Table 7. Note that sample MSH1 (Table 2) is fairly close to the boundary between leifite *sensu stricto* and  ${}^A\Box {}^B\Box \text{Na}_6 [\text{Be}_2 \text{Al}_2 \text{Si}_{16} \text{O}_{39} \text{F}_2]$ , and there seems to be the potential for a considerable number of additional species in the leifite group.

TABLE 7. END-MEMBER COMPOSITIONS FOR THE LEIFITE STRUCTURE-TYPE

	A-site	B-site	Na-site	$T$ -sites and anions
Leifite	Na	(H <sub>2</sub> O)	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>3</sub> Si <sub>15</sub> O <sub>39</sub> F <sub>2</sub> ]
Telyushenkoite	Cs	□	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>3</sub> Si <sub>15</sub> O <sub>39</sub> F <sub>2</sub> ]
–	K	□	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>3</sub> Si <sub>15</sub> O <sub>39</sub> F <sub>2</sub> ]
–	Na	(H <sub>2</sub> O)	Na <sub>6</sub>	[Be <sub>2</sub> Zn <sub>1.5</sub> Si <sub>16.5</sub> O <sub>39</sub> F <sub>2</sub> ]
–	Cs	□	Na <sub>6</sub>	[Be <sub>2</sub> Zn <sub>1.5</sub> Si <sub>16.5</sub> O <sub>39</sub> F <sub>2</sub> ]
–	K	□	Na <sub>6</sub>	[Be <sub>2</sub> Zn <sub>1.5</sub> Si <sub>16.5</sub> O <sub>39</sub> F <sub>2</sub> ]
–	□	□	Na <sub>6</sub>	[Be <sub>2</sub> Al <sub>2</sub> Si <sub>16</sub> O <sub>39</sub> F <sub>2</sub> ]
–	□	□	Na <sub>6</sub>	[Be <sub>2</sub> Zn Si <sub>17</sub> O <sub>39</sub> F <sub>2</sub> ]

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