

FLUORCALCIOPYROCHLORE, A NEW MINERAL SPECIES FROM BAYAN OBO, INNER MONGOLIA, P.R. CHINA

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ABSTRACT

Fluorcalciopyrochlore, ideally $(\text{Ca},\text{Na})_2\text{Nb}_2\text{O}_6\text{F}$, cubic, is a new mineral species (IMA2013-055) occurring in the Bayan Obo Fe-Nb-REE deposit, Inner Mongolia, People's Republic of China. The mineral is found in a dolomite-type niobium rare-earth ore deposit. Associated minerals are dolomite, aegirine, riebeckite, diopside, fluorite, baryte, phlogopite, britholite-(Ce), bastnäsite-(Ce), zircon, magnetite, pyrite, fersmite, columbite-(Fe), monazite-(Ce), rutile, and others. Crystals mostly form as octahedra $\{111\}$, dodecahedra $\{110\}$, and cubes $\{100\}$, or combinations thereof, and generally range in size from 0.01 to 0.3 mm. It is brownish-yellow to reddish-orange in color with a light yellow streak. Crystals of fluorcalciopyrochlore are translucent to transparent with an adamantine to greasy luster on fractured surfaces. It has a conchoidal fracture. No parting or cleavage was observed. The Mohs hardness is 5, and the calculated density is $4.34(1) \text{ g/cm}^3$. The empirical formula is $(\text{Ca}_{1.14}\text{Na}_{0.74}\text{Ce}_{0.06}\text{Sr}_{0.03}\text{Th}_{0.01}\text{Fe}_{0.01}\text{Y}_{0.01}\text{La}_{0.01}\text{Nd}_{0.01})_{\Sigma 2.02}(\text{Nb}_{1.68}\text{Ti}_{0.29}\text{Zr}_{0.02}\text{Sn}_{0.01})_{\Sigma 2.00}\text{O}_{6.00}(\text{F}_{0.92}\text{O}_{0.08})_{\Sigma 1.00}$ on the basis of 7(O,F) anions *pfu*. The simplified formula is $(\text{Ca},\text{Na})_2\text{Nb}_2\text{O}_6\text{F}$. The strongest four reflections in the X-ray powder-diffraction pattern [d in Å (I) hkl] are: 6.040 (9) 1 1 1, 3.017 (100) 2 2 2, 2.613 (17) 0 0 4, 1.843 (29) 0 4 4, and 1.571 (15) 2 2 6. The unit-cell parameters are a 10.4164(9) Å, V 1130.2(2) Å³, Z = 8. The structure was solved and refined in space group $Fd\bar{3}m$ with R = 0.05. The type material is deposited in the Geological Museum of China, Beijing, People's Republic of China, catalogue number M12182.

Keywords: Fluorcalciopyrochlore, new mineral species, pyrochlore supergroup, crystal structure, Inner Mongolia, China.

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INTRODUCTION

The minerals of the pyrochlore supergroup (Atencio *et al.* 2010) show large variations in chemical composition following the general formula $A_2B_2X_6Y$, where the *A* site may be occupied by Na, Ca, Mn, Fe^{2+} , Sr, Ba, Pb, rare-earth elements (*REE*), Bi, Th, and U; the *B* site by Nb, Ta, Ti, Sb, and W; the *X* site by O and OH; and the *Y* site by O, OH, and F. Five groups have been defined on the basis of the atomic proportions of the *B*-site atoms Nb, Ta, Sb, Ti, and W. These groups are pyrochlore, microlite, roméite, betafite, and elsmoreite, respectively. Each of these five groups is comprised of different mineral species depending on the domination of anions and vacancies at the *Y* site as the first prefix, and the cations, H_2O , or vacancies “□” at the *A* site as the second prefix. The F-rich pyrochlore supergroup minerals approved by the IMA-CNMNC are fluorcalciomicrolite $(Ca,Na,□)_2Ta_2O_6F$ (Andrade *et al.* 2013), fluornatromicrolite $(Na,Ca,Bi)_2Ta_2O_6F$ (Witzke *et al.* 2011), and fluornatropyrochlore $(Na,Pb,Ca,REE,U)_2Nb_2O_6F$ (Yin Jingwu *et al.* 2015). In this work we present a new mineral with Nb dominant at the *B* site, F dominant at the *Y* site, and Ca dominant at the *A* site, which gives the name fluorcalciopyrochlore.

A F-rich pyrochlore was first mentioned by Hogarth (1961). There are a few minerals with similar compositions known in the literature, *e.g.*, Ohnenstetter & Piantone (1992), Nasraoui *et al.* (1999), Nasraoui & Bilal (2000), Seifert *et al.* (2000), Thompson *et al.* (2002), Bonazzi *et al.* (2006), and Lee Mi Jung *et al.* (2006). However, earlier F-rich pyrochlores appear in some of the literature with only chemical analytical data. The materials studied were not sufficiently well characterized to grant them official status as a distinct mineral species. Fluorcalciopyrochlore was listed as a possible new species by Christy & Atencio (2013).

In this paper, we describe the properties of fluorcalciopyrochlore, which is a new species of the pyrochlore group (pyrochlore supergroup) discovered in Inner Mongolia. The simplified formula of the mineral is $(Ca,Na)_2Nb_2O_6F$, and the *A* site is mainly occupied by Ca, the *B* site by Nb, and the *Y* site by F. The name was given according to the current IMA-CNMNC-approved nomenclature for the pyrochlore supergroup (Atencio *et al.* 2010). The new mineral and name fluorcalciopyrochlore have been approved by the IMA-CNMNC (IMA2013-055). The holotype specimens are deposited in the Geological Museum of China in Beijing (Xisi Yangrou hutong No. 15, Xicheng District, Beijing), specimen No. M12182.

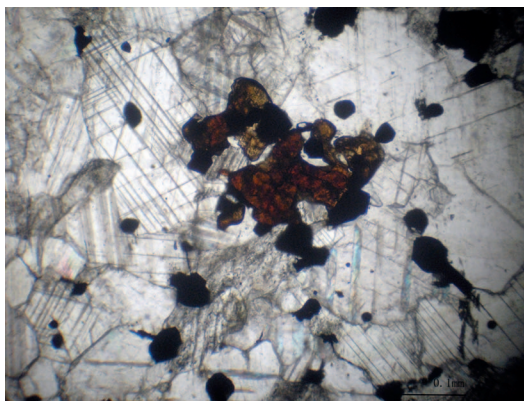


Fig. 1. Fluorcalciopyrochlore as found in carbonatite.

OCCURRENCE AND PARAGENESIS

Fluorcalciopyrochlore occurs in the Bayan Obo Fe-Nb-REE deposit, Inner Mongolia, People's Republic of China (long. $109^{\circ}58.17'$ E, lat. $41^{\circ}47.8'$ N). The Bayan Obo deposit is the world's largest known rare earth element (REE) ore deposit and a major iron and niobium producer as well. The Bayan Obo orebodies are in middle Proterozoic dolomite that is part of a 2000 m thick sequence of quartzite, carbonate rock, and shale. These sedimentary rocks are thought to represent a platform sequence deposited in east-trending grabens along the north edge of the Archean crystalline Sino-Korean craton.

The latest research shows that the Bayan Obo Fe-Nb-REE ore deposit is related to carbonatites (*i.e.*, carbonate rock labeled H8) (Wang Xibin 2002). Enrichment of REE, Nb, and Fe constituent minerals occurs in dolomite, alteration wallrock (aegirine feldspar rock), and in rocks from the late hydrothermal stage. The dolomite is either a dolomitized sedimentary limestone subsequently mineralized and tectonically thrust and folded, or a dolomite (or dolomitized) carbonatite intrusion with late-stage recrystallization and mineralization that has been subsequently tectonically deformed. As a result, the Bayan Obo deposit is not a single type of deposit, but is a complex deposit which includes REE-Nb-Fe carbonate magmatic deposits, replacement alteration deposits, and hydrothermal deposits. It can be referred to as a “Bayan Obo-type deposit” (Wang Kaiyi *et al.* 2012). The carbonatites and the mineralization period of the rare earth elements is 1.3 ~ 1.4Ga (Wang Kaiyi *et al.* 2012). Fluorcalciopyrochlore occurs in the carbonatites, disseminated as fine grains and veinlets or scattered between dolomite crystals (Fig. 1). The mineral is often euhedral or subhedral, associated with dolomite and calcite. The microstructure shows that

TABLE 1. INDEX OF REFRACTION AND COLOR INDEX

R	λ /nm	R	λ /nm
18.73	400	19.56	560
18.83	420	19.67	580
18.93	440	19.71	589 (COM)
19.04	460	19.77	600
19.09	470 (COM)	19.87	620
19.15	480	19.98	640
19.25	500	20.03	650 (COM)
19.35	520	20.08	660
19.46	540	20.19	680
19.49	546 (COM)	20.29	700

there is a symbiosis of fluorcalciopyrochlore with dolomite or calcite and that they crystallized at the same time. The mineral was perhaps a product of bimetatomatism among Ca-Mg-carbonate rock or carbonatite and REE-, F-rich postmagmatic hydrothermal solutions. Other associated minerals found in the carbonate rocks are aegirine, riebeckite, diopside, fluorite, baryte, phlogopite, britholite-(Ce), bastnäsite-(Ce), zircon, magnetite, pyrite, fersmite, columbite-(Fe), monazite-(Ce), and rutile.

APPEARANCE AND PHYSICAL PROPERTIES

While most often fluorcalciopyrochlore occurs as subhedral or allotriomorphic grains, it sometimes forms as octahedra {111}, dodecahedra {110}, and cubes {100}, or combinations thereof (Figs. 1 and 2). Crystals generally range in size from 0.01 to 0.3 mm. It is brownish-yellow to reddish-orange in color with a light yellow streak. Crystals of fluorcalciopyrochlore are translucent to transparent with an adamantine to greasy luster on fractured surfaces. It has a conchoidal fracture. No parting or cleavage was observed. The calculated density is 4.34(1) g/cm³. The average Vickers microhardness is 424 kg/mm² and 5 on the Mohs hardness scale. The mineral appears to be brown and isotropic in thin section (Fig. 1), and the refractivity is greater than 1.9. The calculated refractivity is 2.06 using the Gladstone-Dale relationship $N = Kd + 1$ (K from Mandarino 1981, d: density). Fluorcalciopyrochlore appears grey under reflected light. $R(500) = 19.25\%$. The measured index of refraction values and the color index for fluorcalciopyrochlore are listed in Table 1. The mineral does not dissolve in HCl or HNO₃.

CHEMICAL COMPOSITION

Quantitative chemical analyses were obtained using an electron microprobe (JXA-8100) with an

TABLE 2. ANALYTICAL DATA FOR FLUORCALCIOPYROCHLORE

Constituent	wt.%	Range	SD	EMPA standard
Na ₂ O	6.30	5.40–6.86	0.42	Albite
CaO	17.59	17.16–18.26	0.33	CaSiO ₃
FeO	0.10	0.00–0.20	0.05	Synth-Fe
SrO	0.85	0.54–1.11	0.17	Celestite
PbO	0.24	0.15–0.36	0.06	Crocoite
Ce ₂ O ₃	2.51	1.69–4.07	0.71	Monazite
La ₂ O ₃	0.50	0.36–0.64	0.09	Monazite
Nd ₂ O ₃	0.57	0.26–0.87	0.21	Monazite-(Ce)
Y ₂ O ₃	0.42	0.28–0.66	0.12	Y ₃ Al ₅ O ₁₂
TiO ₂	6.31	5.47–7.95	0.71	Rutile
UO ₂	0.26	0.00–0.43	0.12	Uranium metal
Nb ₂ O ₅	61.36	57.71–65.21	2.02	KNbO ₃
F	4.76	4.54–5.17	0.21	Fluorapatite
ThO ₂	0.76	0.33–1.08	0.23	ThO ₂
ZrO ₂	0.51	0.00–1.14	0.36	ZrO ₂
SnO ₂	0.30	0.24–0.47	0.07	SnO ₂
2F = O	-2.00			
TOTAL	101.44			

Ta₂O₅, MnO below detection limit.

accelerating voltage of 20 kV, a 10 nA beam current, and a beam diameter of 2 μ m (WDS mode). Multiple element standards were used for calibration. The results, based on a 10-point analysis of five grains, are given in Table 2. The uranium content is very low, with UO₂ less than 0.43 wt.%. The empirical formula of fluorcalciopyrochlore, based on 7 anions *pfu* at, is (Ca_{1.14}Na_{0.74}Ce_{0.06}Sr_{0.03}Th_{0.01}Fe_{0.01}Y_{0.01}La_{0.01}Nd_{0.01}) Σ 2.02(Nb_{1.68}Ti_{0.29}Zr_{0.02}Sn_{0.01}) Σ 2.00O_{6.00}(F_{0.92}O_{0.08})_{1.00}. The simplified formula is (Ca,Na)₂Nb₂O₆F.

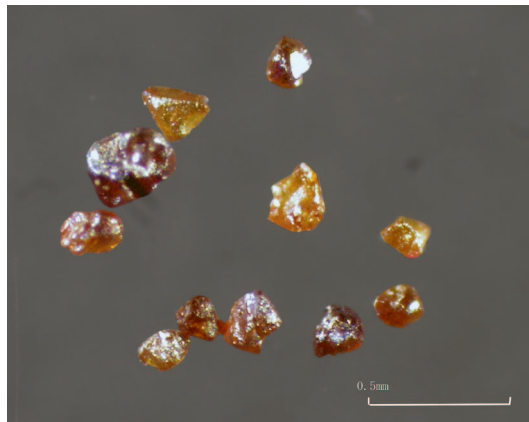


FIG. 2. Grains and crystals of fluorcalciopyrochlore.

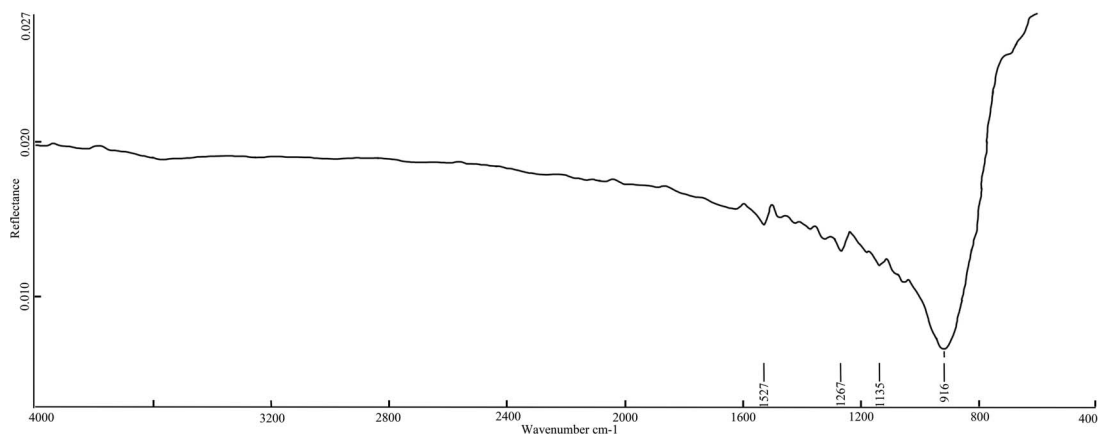


FIG. 3. The infrared spectrum of fluorcalciopyrochlore.

INFRARED SPECTRA

An infrared spectrum was obtained with a Bruker HYPERION 1000 FT-IR Microscope in reflection mode using a single crystal to determine water content. The infrared spectrum is shown in Figure 3. The infrared absorption related to the Nb–O octahedron vibration shows three peaks at 914 cm^{-1} , 1135 cm^{-1} , and the band at 1266 cm^{-1} that represents the vibration of the Ca–O polyhedron. No absorption attributed to OH^- or H_2O was observed.

X-RAY DIFFRACTION AND THE CRYSTAL STRUCTURE

The X-ray powder diffraction (XRPD) data collection was done with a SMART APEX-CCD area detector diffractometer using $\text{MoK}\alpha$ radiation, 45 kV, 35 mA. A new method was used by taking Debye powder diffraction images with a $0.2 \times 0.2 \times 0.1\text{ mm}$ crystal grain and repeated rotation with $\Delta\omega = 10^\circ$. The exposure time was 60 s. Using the method of Li

TABLE 3. X-RAY POWDER DIFFRACTION DATA FOR FLUORCALCIOPYROCHLORE

<i>h</i>	<i>k</i>	<i>l</i>	d_{obs}	d_{calc}	I/I_0
1	1	1	6.040	6.013	9
2	2	2	3.017	3.006	100
0	0	4	2.613	2.604	17
1	1	5	2.008	2.004	1
0	4	4	1.843	1.841	29
2	2	6	1.571	1.570	15
4	4	4	1.503	1.503	2
0	0	8	1.302	1.302	2
2	6	6	1.196	1.194	2
0	4	8	1.165	1.164	1

Guowu *et al.* (2005), very clear Debye ring images were obtained. The powder diffraction pattern and diffraction data were obtained using GADDS software (for WNT 4-1-14, Bruker) and data are reported in Table 3 and Figure 4. The strongest five reflections in the XRPD pattern [d in Å (hkl)] are: 6.0408 (9) 1 1 1, 3.0176 (100) 2 2 2, 2.6131 (17) 0 0 4, 1.8437 (29) 0 4 4, and 1.5711 (15) 2 2 6. The XRPD refined unit-cell parameters are a 10.4285(7) Å, V 1134.1(4) Å³.

Crystal structure data were collected from a single crystal from the original material with a size of $0.2 \times 0.2 \times 0.15\text{ mm}$. The data were obtained with a BRUKER SMART CCD diffractometer using a sealed-tube X-ray source ($\text{MoK}\alpha$ radiation, $\lambda = 0.71073\text{ \AA}$) operating at 45 kV and 35 mA. A hemisphere of intensity data was collected at room temperature. The range of reciprocal space was: h : $\bar{1}6$ –16, k : $\bar{1}5$ –14, l : $\bar{1}5$ –9. A total of 2454 diffraction measurements were collected. The data were corrected for Lorentz-polarization and empirical absorption using the Bruker SADABS program. Diffraction data with $F_o > 3\delta F_o$ were considered as observable points.

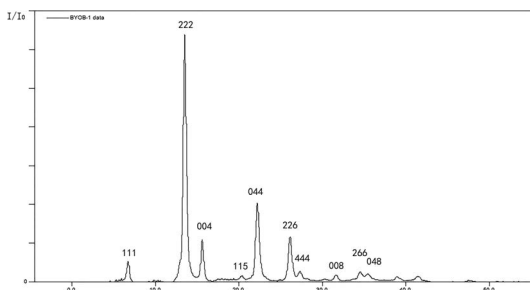


FIG. 4. X-ray powder diffraction pattern of fluorcalciopyrochlore ($\text{MoK}\alpha$).

TABLE 4. THE ATOMIC COORDINATES FOR FLUORCALCIOPYROCHLORE

Atom	Wyck.	x/a	y/b	z/c
Ca*	16d	1	1/2	0
Nb**	16c	3/4	1/2	1/4
O	48f	0.8251(10)	5/8	1/8
F	8b	1/8	5/8	1/8

* Occupancy refined as $Ca_{1.2}$.

** Occupancy refined as $Nb_{0.85}Ti_{0.15}$.

Based on a cubic structure, after the data were merged, there were only 132 independent diffractions [$R(\text{int}) = 0.04$]. These were used for crystal structure determination and refinement. Fluorcalciopyrochlore is cubic and has an $Fd\bar{3}m$ structure with unit-cell parameters a 10.4164(9) Å, V 1130.2(2) Å³, $Z = 8$.

The crystal structure was solved by direct methods using SHELXLS (Sheldrick 2008). The anisotropic and isotropic displacement parameters, occupancies, and atomic coordinates were refined using SHELXTL93. The crystal structure of fluorcalciopyrochlore was refined to $R_1[F^2 > 4\sigma(F^2)] = 0.05$. The atomic parameters are given in Table 4 and anisotropic displacement parameters are given in Table 5.

Fluorcalciopyrochlore belongs to the pyrochlore structure type. The atoms at the B site (Nb,Ti) are located at the center of octahedra forming $[BO_6]$ octahedra. By sharing edges, these octahedra form chains oriented along $[110]$ (Fig. 5). At the A site, the 16d position is occupied mainly by Ca and Na, and is coordinated by six O atoms and two F atoms. The distances from the central Ca ion to the F positions are significantly shorter [2.255(0) Å] than the distances to the O positions [2.590(7) Å] (Table 6).

The occupancy of the A site was done not taking into consideration the smallest heavy atoms, such as Ce. As a result the occupancy for Ca appears higher than it should be.

DISCUSSION

The pyrochlore supergroup minerals present complexities in the specification of endmember formulae. Use of only dominant species at the A site suggests an

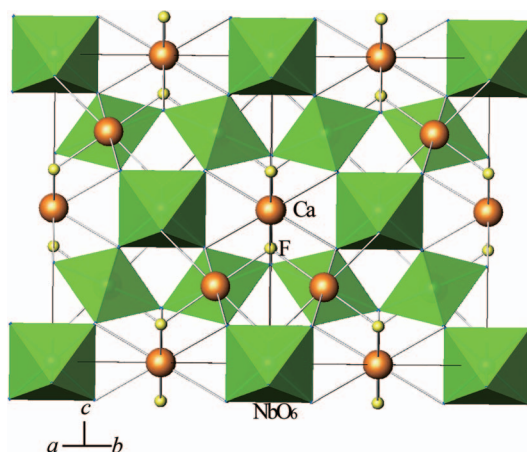


FIG. 5. The crystal structure of fluorcalciopyrochlore viewed along ab ; Ca is orange, Nb is green, and F is yellow.

TABLE 6. SELECTED GEOMETRIC PARAMETERS (Å, °)

Ca–F	2.2552(1)	Nb–O	2.000(4)
Ca–O	2.591(7)		
F–Ca–O	79.97(17)	Nb–O–Ca	105.95(14)

ideal fluorcalciopyrochlore “endmember” formula of $Ca_2Nb_2O_6F$, which is not electrostatically neutral and hence is physically impossible. In this mineral a heterovalent substitution must take place, so from oxycalciopyrochlore, $Ca_2Nb_2O_7$, by simple substitution $(A)Ca^{2+} + (Y)O^{2-} = (A)Na^+ + (Y)F^-$, one can infer the charge-balanced ideal endmember $CaNaNb_2O_6F$. However, rigorous adherence to the principle of using a single dominant species in a dominant-valence group produces a mismatch between species and endmembers in pyrochlores (Atencio *et al.* 2010). The charge-balanced endmember $CaNaNb_2O_6F$ is not at the center of the composition field of a species, but marks the boundary between fluorcalciopyrochlore and fluornatropyrochlore. Conversely, neither of these species names can be associated with a unique charge-balanced endmember. In order to maintain simplicity

TABLE 5. ANISOTROPIC DISPLACEMENT PARAMETERS (Å²) FOR FLUORCALCIOPYROCHLORE

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ca	0.0362(12)	0.0362(12)	0.0362(12)	−0.0051(7)	−0.0051(7)	−0.0051(7)
Nb	0.0238(6)	0.0238(6)	0.0238(6)	0.0014(3)	−0.0014(3)	0.0014(3)
O	0.063(6)	0.032(3)	0.032(3)	0.00000	0.00000	0.010(4)
F	0.048(4)	0.048(4)	0.048(4)	0.00000	0.00000	0.00000

TABLE 7. COMPARISON OF FLUORCALCIOPYROCHLORES FROM VARIOUS OCCURRENCES

Occurrence type	A	B	X	Y	Reference
magmatic carbonatite	$\text{Ca}_{1.14}\text{Na}_{0.74}\text{Ce}_{0.06}\text{Sr}_{0.03}\text{Th}_{0.01}\text{Fe}_{0.01}\text{Y}_{0.01}\text{La}_{0.01}\text{Nd}_{0.01}$	$\text{Nb}_{1.68}\text{Ti}_{0.29}\text{Zr}_{0.02}\text{Sn}_{0.01}$	$\text{O}_{6.08}$	$\text{F}_{0.92}$	This work
phoscorite–carbonatite complex	$\text{Ca}_{1.02}\text{Na}_{0.93}\text{Sr}_{0.05}\text{La}_{0.01}\text{Ce}_{0.03}$	$\text{Nb}_{1.8}\text{Ti}_{0.17}\text{Si}_{0.01}\text{Fe}_{0.02}$	$\text{O}_{5.99}$	$\text{F}_{1.00}$	Lee Mi Jung <i>et al.</i> (2006) Sample: 550R145 No. 4
ultramafic lamprophyre–carbonatite complex	$\text{Ca}_{0.97}\text{Na}_{0.83}\text{Sr}_{0.05}\text{La}_{0.01}\text{Ce}_{0.03}\text{Nd}_{0.01}\text{Th}_{0.01}\text{Fe}_{0.01}$	$\text{Nb}_{1.87}\text{Ti}_{0.13}$	$\text{O}_{6.06}$	$\text{F}_{0.85}$	Seifert <i>et al.</i> (2000)
carbonatite complex	$\text{Ca}_{0.88}\text{Na}_{0.82}\text{Sr}_{0.03}\text{La}_{0.01}\text{Ce}_{0.02}$	$\text{Nb}_{1.86}\text{Ti}_{0.13}$	$\text{O}_{5.80}$	$\text{F}_{0.95}$	Nasraoui <i>et al.</i> (1999)
magmatic unoriented syenite	$\text{Ca}_{1.05}\text{Na}_{0.86}\text{Sr}_{0.03}\text{Ce}_{0.01}$	$\text{Nb}_{1.79}\text{Ti}_{0.14}\text{Ta}_{0.03}\text{Fe}_{0.02}\text{Si}_{0.01}$	$\text{O}_{5.92}$	$\text{F}_{0.97}$	Nasraoui & Bilal (2000) No. 40

and avoid proliferation of names, only the predominant Ca is used to determine fluorcalciopyrochlore species. The endmember of fluorcalciopyrochlore from Bayan Obo can be represented as $(\text{Ca},\text{Na})_2\text{Nb}_2\text{O}_6\text{F}$, in which predominant Ca is used to determine the species, and Na is understood to mean a subordinate amount of an charge-balancing component.

The presence of significant amounts of Ti^{4+} at the B site is charge-balanced by excess Ca^{2+} , according to the substitution $(A)\text{Na}^+ + (B)\text{Nb}^{5+} = (A)\text{Ca}^{2+} + (B)\text{Ti}^{4+}$, which leads to the endmember $\text{Ca}_2\text{NbTiO}_6\text{F}$ (see Table 7). In all the compositions presented, the amount of Ti^{4+} or 4-valent cations at the B site is approximately equal to the content equal to 1–Na.

A small contribution of vacancy may occur at the Y site. This suggests the potential significance of the fourth endmember species in this variety of pyrochlore, namely $\text{Ca}_2\text{Nb}_2\text{O}_6\Box$.

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