Fluorcalciomicrolite, $(Ca,Na,\square)_2Ta_2O_6F$, a new microlite-group mineral from Volta Grande pegmatite, Nazareno, Minas Gerais, Brazil

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ABSTRACT

Fluorcalciomicrolite, $(Ca,Na,\square)_2Ta_2O_6F$, is a new microlite-group, pyrochlore supergroup mineral approved by the CNMNC (IMA 2012-036). It occurs as an accessory mineral in the Volta Grande pegmatite, Nazareno, Minas Gerais, Brazil. Associated minerals include: microcline, albite, quartz, muscovite, spodumene, "lepidolite", cassiterite, tantalite-(Mn), monazite-(Ce), fluorite, "apatite", beryl, "garnet", epidote, magnetite, gahnite, zircon, "tourmaline", bityite, hydrokenomicrolite, and other microlite-group minerals under study. Fluorcalciomicrolite occurs as euhedral, untwinned, octahedral crystals 0.1−1.5 mm in size, occasionally modified by rhombododecahedral faces. The crystals are colourless and translucent; the streak is white, and the lustre is adamantine to resinous. It does not fluoresce under ultraviolet light. Mohs' hardness is 4½−5, tenacity is brittle. Cleavage is not observed; fracture is conchoidal. The calculated density is 6.160 g/cm³. The mineral is isotropic, $n_{calc.}$ = 1.992. The Raman spectrum is dominated by bands of B−X octahedral bond stretching and X−B−X bending modes. The chemical composition (n = 6) is (by wavelength dispersive spectroscopy, H_2O calculated to obtain charge balance, wt.%): Na_2O 4.68, CaO 11.24, MnO 0.01, SrO 0.04, BaO 0.02, SnO_2 0.63, UO_2 0.02, Nb_2O_5 3.47, Ta_2O_5 76.02, F 2.80, H_2O 0.48, O=F −1.18, total 98.23. The empirical formula, based on 2 cations at the B site, is $(Ca_{1.07}Na_{0.81}\square_{0.12})_{\Sigma2.00}(Ta_{1.84}Nb_{0.14}Sn_{0.02})_{\Sigma2.00}[O_{5.93}(OH)_{0.07}]_{6.00}[F_{0.79}(OH)_{0.21}]$. The strongest eight X-ray powder-diffraction lines [d in Å$(I)(hkl)$] are: 5.997(59)(111), 3.138(83)(311), 3.005(100)(222), 2.602(29)(400), 2.004(23)(511), 1.841(23)(440), 1.589(25)(533), and 1.504(24)(444). The crystal structure refinement ($R_1 = 0.0132$) gave the following data: cubic, $Fd\bar{3}m$, $a = 10.4191(6)$ Å, $V = 1131.07(11)$ Å³, $Z = 8$.

Introduction

THE new nomenclature system for pyrochlore-supergroup minerals (Atencio *et al.*, 2010; Christy and Atencio, 2013), approved by CNMNC-IMA, is not based on endmembers in the sense of Hawthorne (2002). As demonstrated by Atencio

et al. (2010), in multidimensional coupled solutions, particularly when several different valence groups occur on more than one site, there is not a unique decomposition of the formula into Hawthornian endmembers. The general formula of the pyrochlore-supergroup minerals is $A_2B_2X_6Y$. The new names are composed of two prefixes and one root name (identical to the name of the group). The first prefix refers to the dominant anion (or cation) of the dominant valence [or H_2O or $\square$] in the Y site. The second prefix refers to the dominant cation of the dominant valence [or H_2O or $\square$] in the A site. For example, fluornatromicrolite (Witzke *et al.*, 2011) has F dominant in Y, and Na dominant in A, whilst fluorcalciomicrolite (this paper) has F dominant in Y, and Ca dominant in A. Use of only dominant species on each site suggests the ideal fluornatromicrolite "endmember" formula $Na_2Ta_2O_6F$, which is not electrically neutral and hence is physically impossible. In the real mineral, two heterovalent substitutions (i.e. Ca for Na and Bi^{3+} for Na) take place and neutrality is obtained. The formula is $(Na,Ca,Bi)_2Ta_2O_6F$.

Electron microprobe analyses showing fluorcalciomicrolite composition have been reported earlier (e.g. Lumpkin *et al.*, 1986; Baldwin, 1989; Ohnenstetter and Piantone, 1992; Tindle and Breaks, 1998; Huang *et al.*, 2002; Geisler *et al.*, 2004; Tindle *et al.*, 2005; Chudík *et al.*, 2011), but a complete description is missing. An intermediate member between fluorcalciomicrolite and fluornatromicrolite, close to $NaCaTa_2O_6F$, was studied by Andrade *et al.* (2011).

Fluorcalciomicrolite, $(Ca,Na,\square)_2Ta_2O_6F$, is a new mineral of the microlite group, pyrochlore supergroup (IMA 2012-036) from the Volta Grande pegmatite, Nazareno, Minas Gerais, Brazil. Type material is deposited with the catalogue number DR731 in the Museu de Geociências, Instituto de Geociências, Universidade de São Paulo, Rua do Lago, 05508-080 São Paulo, Brazil.

Occurrence

Fluorcalciomicrolite occurs as an accessory phase in the Volta Grande pegmatite (21º10′08.6″S 44º36′01.3″W), Nazareno, Minas Gerais, Brazil, in association with microcline, albite, quartz, muscovite, spodumene, "lepidolite", cassiterite, tantalite-(Mn), monazite-(Ce), fluorite, "apatite", beryl, "garnet", epidote, magnetite, gahnite, zircon, "tourmaline", bityite, hydrokenomicrolite

(Andrade *et al.*, 2013), and other microlite-group minerals under study (Heinrich, 1964; Francesconi, 1972; Lagache and Quéméneur, 1997). The fluorcalciomicrolite crystals were collected in a heavy minerals concentrate, so the paragenetic position has not been established. Other crystals of different colours, also corresponding to microlite group minerals, occur in the same concentrate. Some of these crystals are formed by the association between hydrokenomicrolite and fluorcalciomicrolite, which may suggest that hydrokenomicrolite is an alteration product of fluorcalciomicrolite. The crystals used for characterization of fluorcalciomicrolite, however, are homogeneous. The pegmatite belongs to the Sn-Ta-rich São João del Rei Pegmatite Province. The Volta Grande granitic pegmatite is associated with Transamazonian granites (Early Proterozoic) hosted by the Archaean greenstone belt of the Rio das Mortes Valley, which is situated at the southern border of the São Francisco Craton in Minas Gerais, Brazil (Lagache and Quéméneur, 1997). The pegmatite bodies, which are usually large (up to 1200 m × 40 m), show a dominant intermediate zone containing spodumene, microcline, albite and quartz, with an irregular border of an aplitic facies surrounded by an extensive metasomatic aureole with "zinnwaldite", phlogopite and holmquistite. The spodumene-rich core zone is continuous or segmented, and also contains lenses of "lepidolite". The main rock type that hosts the pegmatite is amphibole schist. This pegmatite is an example of rare-element granitic pegmatite exceptionally enriched in lithium and rubidium (Lagache and Quéméneur, 1997).

Habit and physical properties

Fluorcalciomicrolite occurs as untwinned octahedra, occasionally modified by rhombododecahedral faces, from 0.1 to 1.5 mm in size (Fig. 1). The crystals are colourless with a white streak. The lustre is adamantine to resinous. The mineral is translucent. It is non-fluorescent under ultraviolet light. Mohs' hardness is 4½−5. The tenacity is brittle. Cleavage was not observed; fracture is conchoidal. The calculated density is 6.160 g/cm³, based on the empirical formula and unit-cell parameters obtained from the single-crystal X-ray diffraction data.

The mineral is isotropic. The refractive index calculated from the Gladstone-Dale relationship based on the empirical formula is $n_{calc.} = 1.992$ (greater than that of available immersion liquids).
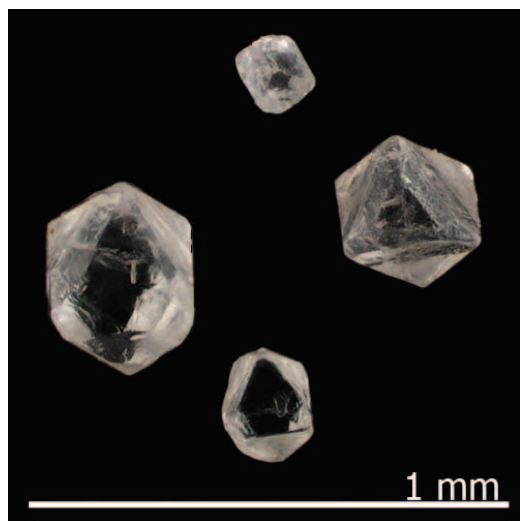
FIG. 1. Fluorcalciomicrolite from Nazareno, Minas Gerais, Brazil (RRUFF project id: R120127).

Composition of fluorcalciomicrolite

The composition of fluorcalciomicrolite was determined using a JEOL JXA 8900RL electron microprobe (wavelength dispersive mode, 20 kV, 20 nA, electron beam diameter 1 μm). The amount of OH was assigned to obtain charge balance of the empirical formula and water was calculated accordingly. The contents of Bi, Ti, Mg and As are below detection limits. Analytical data from six spots are given in Table 1.

The empirical formula, based on 2 cations at the B site is $(Ca_{1.07}Na_{0.81}\square_{0.12})_{\Sigma 2.00}$ $(Ta_{1.84}Nb_{0.14}Sn_{0.02})_{\Sigma 2.00}[O_{5.93}(OH)_{0.07}]_{6.00}$ $[F_{0.79}(OH)_{0.21}]$. The simplified formula is $(Ca,Na,\square)_2Ta_2O_6F$. Following the nomenclature rules (Atencio *et al.*, 2010), the name is fluorcalciomicrolite, because Ca predominates over Na. The formula approaches the charge-balanced endmember $NaCaTa_2O_6F$, which represents the boundary between fluorcalciomicrolite and fluornatromicrolite, however.

Raman data

The Raman spectra of fluorcalciomicrolite (Fig. 2) were collected on a randomly oriented crystal using a Thermo Almega microRaman system, with a 532 nm solid-state laser and a thermoelectrically cooled CCD detector. The laser is partially polarized and spectra were collected with a 4 cm^{-1} resolution and a spot size of ~1 μm (http://rruff.info/R120127).

Factor group analysis yields six Raman-active modes using the site symmetries D_{3d} for A (16d) and B (16c) ions, C_{2v} for X (48f) ions and T_d for Y (8b) ions, respectively (Oliveira *et al.*, 2004). There is one Raman-active vibration associated with the Y-site (8b) ions (F_{2g}) and five for the X-site (48f) ions ($A_g + E_g + 3F_{2g}$) (Glerup *et al.*, 2001). However, the Raman spectrum of fluorcalciomicrolite between 150 and 1180 cm^{-1} shows nine well resolved bands near 168, 187, 292, 331, 417, 505, 664, 792 and 891 cm^{-1}. A tentative assignment of

TABLE 1. Chemical analyses of fluorcalciomicrolite (n = 6).

| Constituent	Wt.%	Range	SD	Probe standard
Na$_2$O	4.68	4.41−4.83	0.13	jadeite
CaO	11.24	11.16−11.30	0.05	andradite
MnO	0.01	n.d.−0.02	0.01	rhodonite
SrO	0.04	0.01−0.06	0.02	celestite
BaO	0.02	n.d.−0.05	0.02	baryte
SnO$_2$	0.63	0.50−0.80	0.10	cassiterite
UO$_2$	0.02	n.d.−0.05	0.03	UO$_2$
Nb$_2$O$_5$	3.47	3.14−3.81	0.21	metallic Nb
Ta$_2$O$_5$	76.02	74.61−77.43	0.20	metallic Ta
F	2.80	2.41−2.95	0.20	fluorite
H$_2$O*	0.48			
O=F	−1.18			
Total	98.23			

* calculated to obtain charge balance.
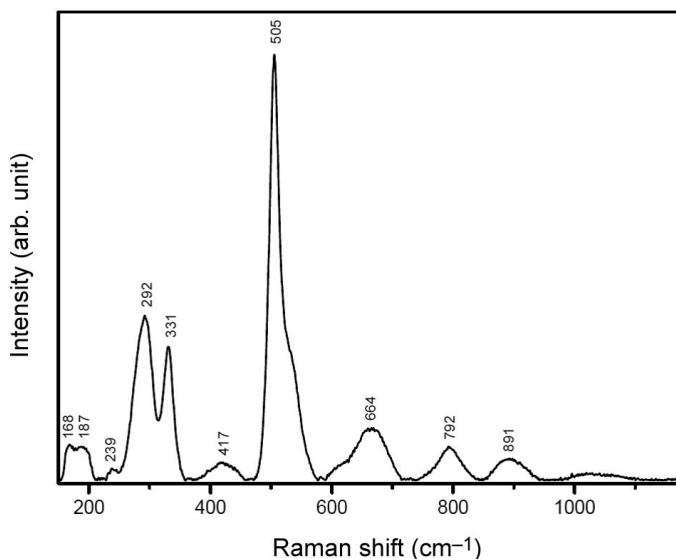
Fɪɢ. 2. Raman spectrum of fluorcalciomicrolite.

observed Raman modes for fluorcalciomicrolite based on previous studies on pyrochlore supergroup minerals (Geisler *et al*., 2004; Oliveira *et al*., 2004; Arenas *et al*., 2010) is given in Table 2.

Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) data were acquired with a Bruker Hyperion 3000 microscope at INFN (Frascati, Rome) using 4 cm^{-1} resolution, 128 scans co-added, detector MCT, beam size 30 μm. The enlarged doublet at

$3600-3581$ cm^{-1} indicates the presence of $(OH)^-$ (Fig. 3).

Crystal structure

X-ray powder diffraction data were obtained using a Siemens D5000 diffractometer equipped with a position-sensitive detector. Data (for Cu$K\alpha$, 40 kV and 40 mA) are given in Table 3. Unit-cell parameters refined from powder data (space group $Fd\bar{3}m$) are $a = 10.417(1)$ Å, $V = 1130.41(19)$ Å^3 and $Z = 8$.

Tᴀʙʟᴇ 2. Tentative assignment of major Raman bands for fluorcalciomicrolite at $T = 296$ K.

| Peak (cm^{-1})	Intensity	Tentative assignment
891	relatively strong, broad	not assigned
792	relatively strong, sharp	not assigned
664	relatively strong, broad	B–X octahedra stretching
530	weak, shoulder	B–X octahedra stretching
505	very strong, sharp	B–X octahedra stretching
417	relatively strong, broad	X–B–X bending
331	strong, sharp	X–B–X bending
292	strong, sharp	X–B–X bending
239	weak, broad	A–Y stretching + X–A–Y bending
187	relatively strong	not assigned
168	relatively strong	not assigned
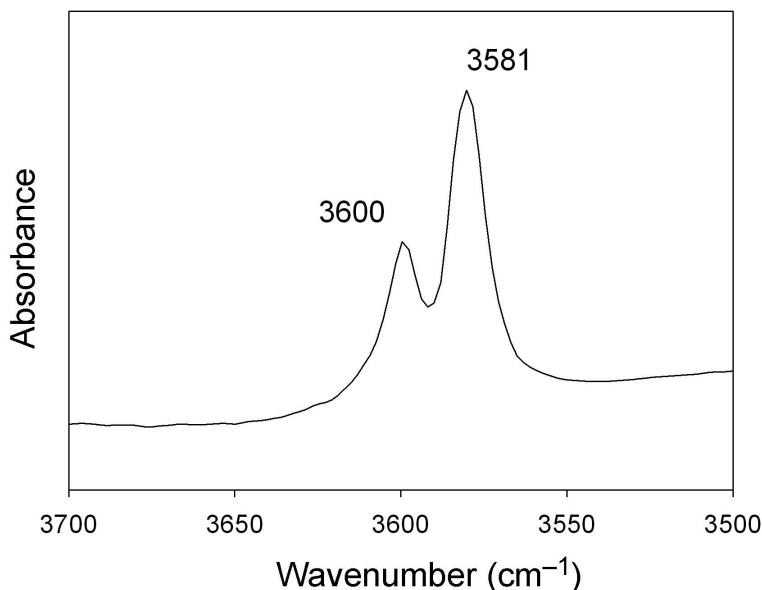
FIG. 3. Fourier transform infrared spectroscopy (FTIR) of fluorcalciomicrolite.

A colourless crystal of dimensions 0.08 mm × 0.07 mm × 0.07 mm was used for the structural investigation. X-ray diffraction measurements were made on a Bruker X8 APEX2 CCD diffractometer with graphite-mono-chromated MoKa (λ = 0.71073 Å) radiation. Data were collected to 75º in 2θ. Final unit-cell parameters were based on 970 reflections with the index ranges $-17 \leqslant h \leqslant 16$, $-7 \leqslant k \leqslant 14$, $-16 \leqslant l \leqslant 17$. The *APEX2* program was used for data collection, and the integration and scaling of the reflections were performed with the *SAINT* system of programs (Bruker, 2004). A SADABS absorption correction was applied (Bruker, 2004). All the structure refinements were performed with the scattering factors for neutral atoms (Ibers and Hamilton, 1974). The structure was solved using direct methods with *SHELXS-97* (Sheldrick, 2008). The standard pyrochlore structures have all atoms occupying special positions (A = 16d, B = 16c, X = 48f and Y = 8b) in $Fd\bar{3}m$ (Subramanian *et al.*, 1983). The A- and B-site occupancies were constrained initially by the microprobe chemical analysis as $(Ca_{1.07}Na_{0.81})_{\Sigma 1.88}$ and $(Ta_{1.84}Nb_{0.16})_{\Sigma 2.00}$, respectively. The amount of Sn was treated as Nb. The Y site was set as fully occupied, and refined using only the scattering curve of F, which is dominant. The refinement showed that the Y position is fully occupied and located at Wyckoff position 8b. The Y occupancy did not present any positional disorder during refinement. Refinement of this model converged to R_1 = 0.0132 and $wR2$ = 0.0267, with maximum and minimum electron-densities of +0.86 and -0.85 $e/Å^3$. The details related to collection procedures and structure refinement are presented

TABLE 3. X-ray powder-diffraction data for fluor-calciomicrolite.

I_{rel}	$d_{meas.}$(Å)	$d_{calc.}$(Å)	h	k	l
59	**5.997**	**6.014**	1	1	1
83	**3.138**	**3.141**	3	1	1
100	**3.005**	**3.007**	2	2	2
29	**2.602**	**2.604**	4	0	0
14	2.389	2.390	3	3	1
23	**2.004**	**2.005**	5	1	1
23	**1.841**	**1.8415**	4	4	0
13	1.760	1.7608	5	3	1
25	**1.589**	**1.5886**	5	3	3
17	1.571	1.5704	6	2	2
24	**1.504**	**1.5036**	4	4	4
21	1.459	1.4587	7	1	1
7	1.356	1.3562	7	3	1

* indexed with a = 10.417(1) Å

TABLE 4. Crystal data and details of structure refinement.

Temperature (K)	293(2)
Crystal colour	colourless
Crystal size (mm)	$0.08 \times 0.07 \times 0.07$
Formula weight	519.46
Crystal system	Cubic
Space group	$Fd\bar{3}m$ (#227, origin at *16c*,–3m)
Unit-cell dimension *a*	10.4191(6) Å
Unit-cell volume *V*	1131.07(11) Å^3
Z	8
Density (calculated)	6.101 g/cm^3
Absorption coefficient	36.05
F(000)	1813
Reflections collected/unique	1418/147
Parameters	14 (R_{int} = 0.0300)
Goodness-of-fit on *F*	1.283
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0132$, $wR_2 = 0.0267$
Largest diff. peak and hole	0.86 and –0.85 e.Å^{-3}

in Table 4. Charge balance was maintained by replacing O by OH at the *X*(48f) and *Y* (8*b*) positions. The refined formula is $(Ca_{1.06}Na_{0.82})_{\Sigma1.88}(Ta_{1.79}Nb_{0.21})_{\Sigma2.00}[O_{5.94}(OH)_{0.06}]_{\Sigma6.00}[F_{0.79}(OH)_{0.21}]$ (Fig. 4). Final atomic positions are given in Table 5 and cell parameters and selected bond lengths (Table 6) are compared with an intermediate member of the fluorcalciomicrolite-fluornatromicrolite solid solution (Andrade *et al.*, 2011) and fluorcalcio-
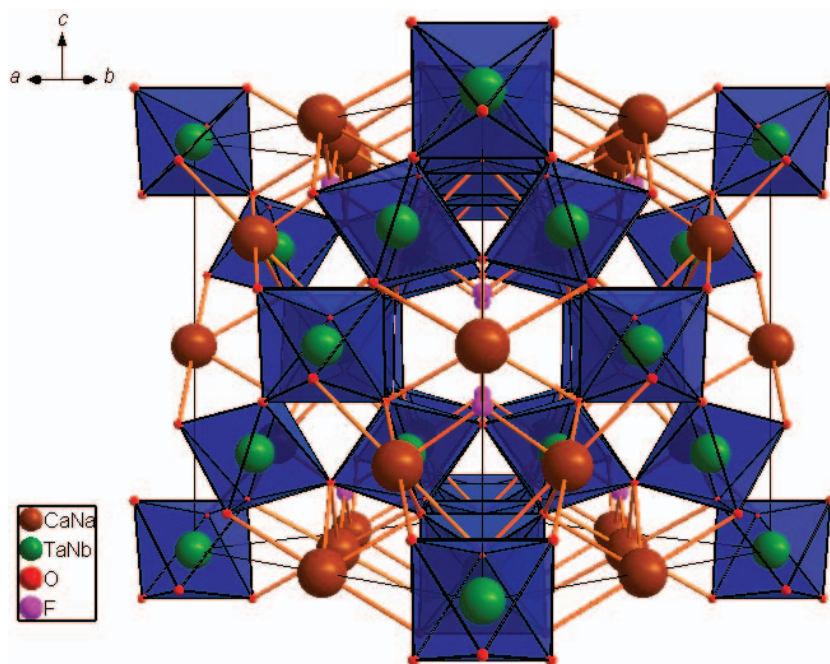

FIG. 4. Fluorcalciomicrolite structure.

TABLE 5. Wyckoff positions, site occupancies, atom coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$) in fluorcalciomicrolite.

	Wyckoff	Occupancy	x	y	z	U_{eq} ($\mathring{A}^2$)
A	16d	0.94	0	0	.5	0.0130(6)
B	16c	1	0	0	0	0.00438(10)
X	48f	1	1/8	1/8	0.3183(3)	0.0087(6)
Y	8b	1	1/8	1/8	5/8	0.0121(10)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
A	0.0130 (6)	0.0130 (6)	0.0130 (6)	−0.0041 (3)	−0.0041 (3)	−0.0041 (3)
B	0.00438 (10)	0.00438 (10)	0.00438 (10)	−0.00062 (4)	−0.00062 (4)	−0.00062 (4)
X	0.0082 (7)	0.0082 (7)	0.0097 (11)	0.0051 (9)	0	0
Y	0.0121(10)	0.0121(10)	0.0121(10)	0	0	0

TABLE 6. Refined crystal-structure data for fluorcalciomicrolite.

	1	2	3	4
a (Å)	10.4191(6)	10.4396(12)	10.421(2)	10.43308(9)
A	$Ca_{1.06}Na_{0.82}\square_{0.12}$	$Ca_{0.88}Na_{0.88}Pb_{0.02}\square_{0.22}$	$Ca_{1.5}$*	$Ca_{1.5}$
B	$Ta_{1.79}Nb_{0.21}$	$Ta_{1.70}Nb_{0.14}Si_{0.12}As_{0.04}$	Ta_2	Ta_2
X	$O_{5.94}(OH)_{0.06}$	$O_{5.75}(OH)_{0.25}$	O_6	O_6
Y	$F_{0.79}(OH)_{0.21}$	$F_{0.73}\square_{0.27}$	F	F
$A(16d)–X(48f) \times 6$	2.6415 (19)	2.646(7)	2.649(8)	2.636(9)
$A(16d)–Y(8b) \times 2$	2.2558(1)	2.2602(2)	2.256(8)	2.259(1)
$B(16c)–X(48f) \times 6$	1.9744 (10)	1.979(3)	1.971(6)	1.982(6)

1 Nazareno, Minas Gerais, Brazil (this work)
2 Coronel Murta, Minas Gerais, Brazil (Andrade *et al.*, 2011),
3 Synthetic (Siegrist *et al.*, 1997),
4 Synthetic (Le Berre *et al.*, 2007),
* Erroneously expressed as Ca_2

microlite synthetic compounds (Siegrist *et al.*, 1997; Le Berre *et al.*, 2007).

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