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RUIFRANCOITE, A NEW Fe^{3+}-DOMINANT MONOCLINIC MEMBER OF THE ROSCHERITE GROUP FROM GALILÉIA, MINAS GERAIS, BRAZIL

Daniel ATENCIO[§]

Instituto de Geociências, Universidade de São Paulo, Rua do Lago, 562, 05508-080, São Paulo, SP, Brazil

Nikita V. CHUKANOV

Institute of Problems of Chemical Physics, Chernogolovka, Moscow Oblast, 142432, Russia

José M.V. COUTINHO

Instituto de Geociências, Universidade de São Paulo, Rua do Lago, 562, 05508-080, São Paulo, SP, Brazil

Luiz A.D. MENEZES FILHO

Rua Esmeralda, 534, Prado, 30410-080, Belo Horizonte, MG, Brazil

Viktor T. DUBINCHUK

All-Russian Scientific-Research Institute of Mineral Resources (VIMS), Staromonetny per. 31, Moscow 119017, Russia

Steffen MÖCKEL

Alpha-Geophysik in Sachsen, Neudorfer Str. 18, D–09629 Burkersdorf, Germany

ABSTRACT

Ruifrancoite, $Ca_2(\square,Mn^{2+})_2(Fe^{3+},Mg,Mn^{2+},Fe^{2+},Al)_4Be_4(PO_4)_6(OH)_4(OH,H_2O)_2{\bullet}4H_2O$, monoclinic, is a newly recognized member of the roscherite group. It occurs as a late vug-filling mineral in albite and muscovite, closely associated with cyrilovite and meurigite in a granite pegmatite at the Sapucaia (Proberil) mine, Sapucaia do Norte, Galiléia County, Minas Gerais, Brazil. Ruifrancoite occurs as needles or laths 1 to 2 μm thick and up to 100 μm long in fan-shaped botryoidal arrangements. The color is reddish brown, the streak is orange-brown, and the luster is vitreous. Ruifrancoite is transparent and non-fluorescent. The measured density is 2.88(1) g/cm^3 (by the hydrostatic weighing method), and the calculated density is 2.859 g/cm^3. The mineral is biaxial (+), α 1.665(3), β 1.665(3), γ 1.682(3) (white light), $2V$(obs.) 0–10°, $2V$(calc.) 0°. The orientation is $X = b$, $Y \wedge c \approx 16°$; dispersion $r > v$, very strong. Pleochroism is γ (brownish red) $> \alpha = \beta$ (light brownish red). Infrared and Mössbauer spectroscopic data are given. The chemical composition is (wt.%, EDS mode, electron microprobe, $Fe^{2+}:Fe^{3+}$ ratio by Mössbauer data): CaO 9.81, MgO 3.23, MnO 8.10, FeO 3.93, Fe_2O_3 12.51, Al_2O_3 0.86, BeO (by ICP–AES) 8.41, P_2O_5 39.46, H_2O (by Penfield method) 12.56, total 98.87. The empirical formula, based on six (PO_4) groups, with calculated BeO of 9.23%, is $Ca_{1.89}(\square_{1.45}Mn^{2+}_{0.55})_{\Sigma 2.00}(Fe^{3+}_{1.69}Mg_{0.86}Mn^{2+}_{0.68}Fe^{2+}_{0.59}Al_{0.18})_{\Sigma 4.00}\ Be_{4.00}(PO_4)_6\ [(OH)_{4.75}(H_2O)_{1.25}]{\bullet}3.90H_2O$. The ideal end-member formula of ruifrancoite is $Ca_2\square_2Fe^{3+}_4Be_4(PO_4)_6(OH)_6{\bullet}4H_2O$. The strongest reflections of the powder-diffraction pattern [d in Å(I)(hkl)] are: 9.485(44)($\bar{1}10$), 5.943(100)(020), 4.821(65)(310), 3.176(44)(330), 2.784(41)(240), and 2.643(42)(600). The unit-cell parameters refined from powder data are a 15.911(7), b 11.894(7), c 6.625(7) Å, β 94.5(1)°, V 1250(1) Å^3, $Z = 2$. The space group is $C2/c$. The mineral is named in honor of Professor Rui Ribeiro Franco (b. 1916), in recognition of his outstanding contributions to Brazilian mineralogy and geology. Former descriptions of "roscherite" at Sapucaia mine, and "Fe^{3+}-dominant species" at Pomarolli farm, Linópolis, Divino das Laranjeiras, Minas Gerais, Brazil, should be considered to refer to ruifrancoite.

Keywords: ruifrancoite, new mineral species, phosphate, roscherite group, Sapucaia mine, Galiléia, Minas Gerais, Brazil.

[§] *E-mail address*: datencio@usp.br

SOMMAIRE

La ruifrancoïte, de composition idéale $Ca_2(\square,Mn^{2+})_2(Fe^{3+},Mg,Mn^{2+},Fe^{2+},Al)_4Be_4(PO_4)_6(OH)_4(OH,H_2O)_2{\bullet}4H_2O$, est un nouveau membre du groupe de la roscherite. On l'a trouvé dans des poches d'une pegmatite granitique exploitée à la mine Sapucaia (Proberil), Sapucaia do Norte, comté de Galiléia, Minas Gerais, au Brésil, en association avec albite, muscovite, cyrilovite et meurigite.La ruifrancoïte se présente en aiguilles ou en plaquettes entre 1 et 2 μm d'épaisseur et jusqu'à 100 μm de long, agencées en amas botryoïdaux. Elle est brun rougeâtre, avec une rayure orange-brun, et l'éclat est vitreux. Elle est transparente et non-fluorescente. La densité mesurée est 2.88(1) g/cm^3 (mesurée par la méthode hydrostatique), et la densité calculée est 2.859 g/cm^3. Il s'agit d'un minéral biaxe (+), α 1.665(3), β 1.665(3), γ 1.682(3) (lumière blanche), 2V(obs.) 0–10°, 2V(calc.) 0°. L'orientation est $X = b$, $Y \wedge c \approx 16°$; dispersion $r > v$, très intense. Elle est pléochroïque: γ (rouge brunâtre) > $\alpha = \beta$ (rouge brunâtre pâle). Nous présentons les spectres infrarouge et de Mössbauer. La composition chimique (résultats d'analyses par dispersion d'énergie avec une microsonde électronique, exprimés en % pondéraux, avec rapport Fe^{2+}:Fe^{3+} selon le spectre de Mössbauer): CaO 9.81, MgO 3.23, MnO 8.10, FeO 3.93, Fe_2O_3 12.51, Al_2O_3 0.86, BeO (par ICP–AES) 8.41, P_2O_5 39.46, H_2O (par la méthode de Penfield) 12.56, pour un total de 98.87. La formule empirique, fondée sur six groupes (PO_4) et calculée une teneur en BeO de 9.23%, est $Ca_{1.89}(\square_{1.45}Mn^{2+}_{0.55})_{\Sigma 2.00}$ $(Fe^{3+}_{1.69}Mg_{0.86}Mn^{2+}_{0.68}Fe^{2+}_{0.59}Al_{0.18})_{\Sigma 4.00}$ $Be_{4.00}(PO_4)_6$ $[(OH)_{4.75}(H_2O)_{1.25}]{\bullet}3.90H_2O$. La formule du pôle idéal serait $Ca_2\square_2Fe^{3+}_4Be_4(PO_4)_6(OH)_6{\bullet}4H_2O$. Les raies les plus intenses du spectre de diffraction (méthode des poudres) [d en Å(I)(hkl)] sont: 9.485(44)($\bar{1}10$), 5.943(100)(020), 4.821(65)(310), 3.176(44)(330), 2.784(41)(240), et 2.643(42)(600). Les paramètres de la maille élémentaire ont été affinés à partir des données sur poudre: a 15.911(7), b 11.894(7), c 6.625(7) Å, β 94.5(1)°, V 1250(1) Å^3, $Z = 2$, groupe spatial $C2/c$. Le nom choisi honore le professeur Rui Ribeiro Franco (né en 1916) pour ses contributions majeures à la minéralogie et la géologie du Brésil. Les descriptions antérieures de la "roscherite" à la mine Sapucaia, et d'une espèce à dominance de Fe^{3+} à la ferme Pomarolli, Linópolis, Divino das Laranjeiras, Minas Gerais, Brésil, seraient attribuables à la ruifrancoïte.

(Traduit par la Rédaction)

Mots-clés: ruifrancoïte, nouvelle espèce minérale, phosphate, groupe de la roscherite, mine Sapucaia, Galiléia, Minas Gerais, Brésil.

INTRODUCTION

Ruifrancoite, ideally $Ca_2(\square,Mn^{2+})_2(Fe^{3+},Mg,Mn^{2+},Fe^{2+},Al)_4Be_4(PO_4)_6(OH)_4(OH,H_2O)_2{\bullet}4H_2O$, is a new monoclinic member of the roscherite group. It was discovered in a granitic pegmatite at the Sapucaia (Proberil) mine, Sapucaia do Norte, Galiléia County, Minas Gerais, Brazil. The mineral is named after Professor Rui Ribeiro Franco (b. 1916), in recognition of his outstanding contributions to Brazilian mineralogy and geology. We cite in particular the three-volume treatise *Minerais do Brasil* (Franco *et al.* 1972), and several papers, chapters and books on mineralogy, geochemistry, gemology, petrography, petrology and geology (*e.g.,* Franco 1981). The new mineral species has been approved by the CNMMN (IMA 2005–061).

The holotype sample is deposited under the number DR587 in the Museu de Geociências at the Instituto de Geociências, Universidade de São Paulo. The cotype material (used for Mössbauer spectroscopy) is deposited in the Mineralogical Collection of the Technische Universität Bergakademie Freiberg with the inventory number 81420.

BACKGROUND INFORMATION

The history of the roscherite group is somewhat complex. Roscherite was first described by Slavík (1914) as a monoclinic hydrous phosphate of calcium, manganese, iron, and aluminum, with Mn > Fe. With results of spectrographic and chemical analyses, Lindberg (1958) showed that beryllium occurs as a major constituent, and aluminum, only in traces. Lindberg (1958) also studied Fe^{2+} and Fe^{3+} analogues of this mineral, but used the name roscherite for the three species. Fanfani *et al.* (1975) described the Mg-dominant species isostructural with roscherite, but also applied the name roscherite. Fanfani *et al.* (1977) described a triclinic Mn-dominant roscherite-group mineral, but did not introduce a new name for it. The name *zanazziite* was created for the Mg-dominant species by Leavens *et al.* (1990). They also cited a triclinic Fe–Mg member of this group. The name *greifensteinite* was introduced for the Fe^{2+} species by Chukanov *et al.* (2002). Chukanov *et al.* (2006) described *atencioite*, a triclinic Fe–Mg member of this group. Atencio *et al.* (2008) named the triclinic Mn-dominant roscherite-group mineral *footemineite*. A monoclinic Zn-dominant roscherite-group mineral was named *guimarãesite* by Chukanov *et al.* (2007). In this paper, we describe yet a new species, ruifrancoite, the monoclinic Fe^{3+}-dominant member of the group, probably the same as the one first studied by Lindberg (1958).

OCCURRENCE

Ruifrancoite occurs as a late vug-filling product in a zoned, LCT-type granitic pegmatite. It crystallized on albite and muscovite and occurs in close association with cyrilovite, $NaFe^{3+}_3(PO_4)_2(OH)_4{\bullet}2H_2O$, and meurigite, $KFe^{3+}_7(PO_4)_5(OH)_7•8H_2O$. The Sapucaia mine has been an important producer of muscovite and beryl, extracted from various zones of the oval-shaped pegmatite (Pecora *et al.* 1950). In the core zone, essentially composed of quartz, perthite, muscovite, and beryl, there is an irregular body of triphylite weighing several hundred tonnes, which was partially altered by hydrothermal fluids and later, as a result of weathering. An assemblage of rare phosphates resulted, six of which have been described as new mineral species: frondelite, $(Mn,Fe^{2+})Fe^{3+}_4(PO_4)_3(OH)_5$ (Lindberg 1949), faheyite, $Be_2(Mn,Mg,Na)Fe^{3+}_2(PO_4)_4•6H_2O$ (Lindberg & Murata 1952, 1953), moraesite, $Be_2PO_4(OH)•4H_2O$ (Lindberg *et al.* 1953), barbosalite, $Fe^{2+}Fe^{3+}_2(PO_4)_2(OH)_2$, tavorite, $LiFe^{3+}PO_4(OH)$ (Lindberg & Pecora 1954, 1955), and arrojadite-(PbFe), $[PbFe^{2+}Na_2Ca(Fe^{2+},Mn,Mg)_{13}Al(PO_4)_{11}(PO_3OH)(OH,F)_2$ (Chopin *et al.* 2006). Detailed studies of the phosphate mineral associations (Lindberg & Pecora 1958, Cassedanne & Baptista 1999) have revealed three principal assemblages corresponding to separate geochemical environments: (1) the triphylite assemblage, as fracture fillings and replacements in massive triphylite, (2) the frondelite assemblage, consisting of residual phosphates enclosed by frondelite, results of a massive replacement of that mineral, and minerals on walls of vugs in frondelite, and (3) the silicate assemblage, formed by secondary crusts on altered albite, products of the alteration of beryl, and replacement and vug fillings in muscovite. Ruifrancoite occurs in the third assemblage.

APPEARANCE AND PHYSICAL PROPERTIES

Ruifrancoite occurs as needles and laths with a thickness of 1 to 2 μm and a length of about 100 μm in fan-shaped botryoidal arrangements (Figs. 1, 2). Its color is reddish brown, the streak is orange-brown, and the luster is vitreous. The mineral is transparent and non-fluorescent. The Mohs hardness is probably about 4½, by analogy with other roscherite-group minerals. The crystals are not adequate for morphological observations because of their habit and size. They are apparently bound by two pinacoids, {010} and {100}, and a prism. By analogy with other roscherite-group minerals, ruifrancoite probably has a perfect cleavage on {100} and a good cleavage on {010}. The mineral is brittle. Measured density is 2.88(1) g/cm^3 (with the hydrostatic weighing method), and the calculated density is 2.859 g/cm^3.

The monoclinic Fe^{3+}-dominant roscherite-group mineral described earlier from Sapucaia mine under the name "roscherite" by Lindberg (1958) and Lindberg & Pecora (1958), probably ruifrancoite, occurs as brown, prismatic single crystals, crystal aggregates, and granular masses, in vugs in muscovite, and as crusts on beryl. The mineral is characterized by well-developed faces of the forms {111}, {010}, and {100}. On a few crystals, small faces of the forms {102}, {201}, $\{\bar{1}01\}$, $\{\bar{2}01\}$, $\{\bar{3}01\}$, and $\{\bar{1}11\}$ are present, but are of poor quality. The crystals are tabular parallel to (100).

OPTICAL PROPERTIES

The type specimen of ruifrancoite is biaxial (+), α 1.665(3), β 1.665(3), γ 1.682(3) (white light), 2*V*(obs.) 0–10°, 2*V*(calc.) 0°. Orientation is X = *b*, Y ^ *c* ≈16°; dispersion $r > v$, very strong. The pleochroic scheme is γ brownish red > α = β light brownish red. Optical data for other samples of ruifrancoite are presented in Table 1.

INFRARED SPECTROSCOPY

A sample of ruifrancoite weighing 1 mg was mixed with 150 mg of anhydrous KBr, pelletized, and analyzed using a Specord 75 IR spectrophotometer. The infrared spectrum of a pure KBr disk was subtracted from the overall spectrum. Polystyrene and gaseous NH_3

TABLE 1. OPTICAL PROPERTIES OF RUIFRANCOITE

	1	2	3	4	5	6	7
Optical sign	Bi(+)	Bi(–)	Bi(+)	Bi(+) or (–)	Bi(+)	Bi(+)	Bi(–)
α	1.665(3)	1.660	1.636	1.670 to 1.680	1.661(5)	n.d.	n.d.
β	1.665(3)	1.665	1.641	1.680 to 1.700	1.663(5)	1.670(4)	1.652(3)
γ	1.682(3)	1.668	1.651	1.695 to 1.705	1.666(3)	1.670(4)	1.660(3)
$2V_z$ obs. (°)	~0-10			60 to 90	~70		~45
$2V_z$ calc. (°)	0	75.3	70.9				

Columns 1 to 3. Sapucaia pegmatite, Galiléia, Minas Gerais. 1. Type specimen of ruifrancoite. 2. Mn-rich ruifrancoite. 3. Ruifrancoite studied by Lindberg (1958), considered as roscherite, dispersion $r > v$, strong; absorption yellow. Where present as a fine-grained replacement of brown frondelite, admixed with multicolored beryl, it has higher indices of refraction, with γ in the range 1.67 to 1.68. Columns 4 to 7. Lavra Pomarolli, Linópolis, Divino das Laranjeiras, Minas Gerais [column 4 from Atencio *et al.* (2005), columns 5 to 7: this paper].

FIG. 1. Botryoidal reddish brown aggregates of ruifrancoite associated with cyrilovite, and yellowish white spheres of meurigite on white mica from Sapucaia mine, Galiléia, Minas Gerais, Brazil.

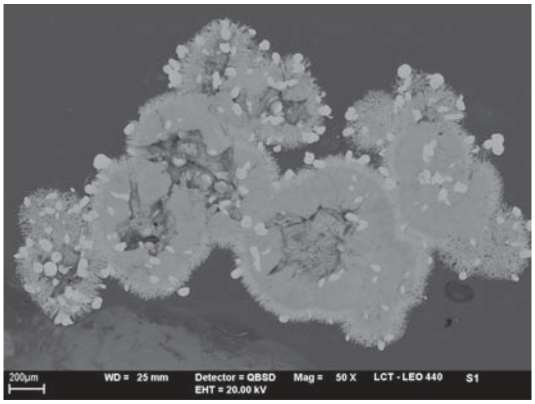

FIG. 2. Back-scattered electron image. The darker radiating aggregates correspond to ruifrancoite, and the lighter-colored grains, to cyrilovite.

were used as frequency standards; the precision of the frequency measurement is ±1 cm^{-1}; the mean resolution for the range 400–1600 cm^{-1} is 0.8 cm^{-1}. Wavenumbers of the bands observed on the infrared spectrum (Fig. 3) are in cm^{-1} (wavenumbers of strongest bands are underlined): 3600, 3580 (stretching vibrations of OH groups), 3450, 3345 (stretching vibrations of H_2O molecules), 1660, 1605, 1525 (bending vibrations of H_2O molecules), 1088, 1015, 970 (stretching vibrations of PO_4^{3-} anions), 819, 760, 722, 700 (stretching vibrations of BeO_4 tetrahedra and bending vibrations of *M*–OH fragments, where *M* is an octahedrally coordinated cation), 611, 559 (bending vibrations of PO_4^{3-} anions), 520, 440 (stretching vibrations of the MO_6 octahedra). Triclinic minerals of the roscherite group (atencioite, footemineite) show a characteristic splitting of the P–O and Be–O stretching bands, whereas monoclinic roscherite-group minerals (roscherite, greifensteinite and zanazziite) do not show such splitting (Chukanov *et al*. 2006). Usually in each type of normal vibrations, coordination polyhedra of one type take part. Here, the IR spectrum does not reflect local vibrations of coordination polyhedra, but rather collective vibrations of areas of a crystal (typical sizes of regions of coherent vibrations are several tens of Å). The increase in the number of non-equivalent coordination polyhedra (due to different causes, like site splitting, solid solution, lowering of symmetry) results in the increase of the number of normal vibrations active in the IR spectrum. But this connection is not simple owing to resonance interactions. In the case of roscherite-group minerals, the lowering of symmetry results in band splitting. Splitting of the bands in roscherite-group minerals is empirically observed.

MÖSSBAUER SPECTROSCOPY

The Mössbauer spectrum has been obtained using a modified WISSEL spectrometer, simultaneously recording in 512 channels, with signal accumulation

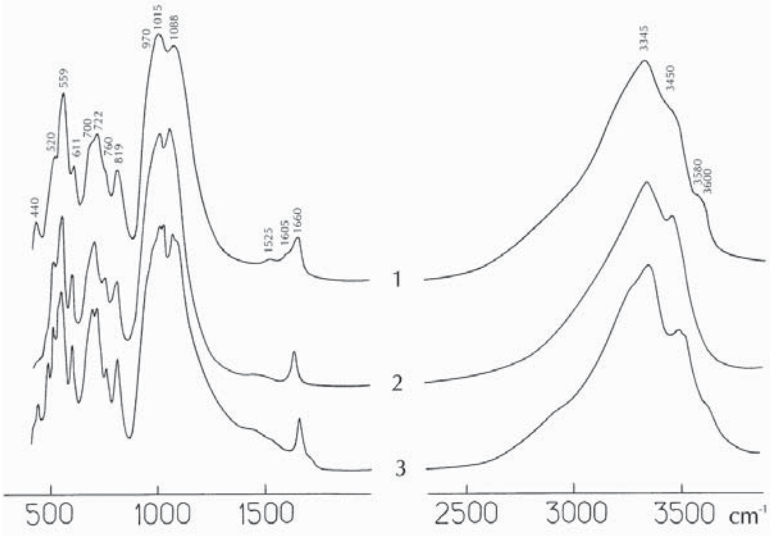

FIG. 3. IR spectrum of ruifrancoite (1). For comparison, IR spectra of other iron-rich roscherite-group minerals are given. Greifensteinite (2) is monoclinic, and atencioite (3) is triclinic.

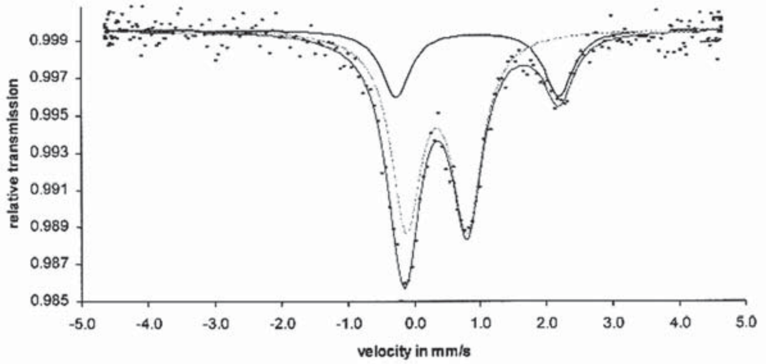

FIG. 4. The Mössbauer spectrum of ruifrancoite.

over 40 hours. The Mössbauer spectroscopic data (Fig. 4) gave a Fe^{3+}/Fe^{2+} value of 74.2/25.8. Total Fe was divided over Fe_2O_3 and FeO according to these results. There is only one Fe^{3+} doublet in the Mössbauer spectrum. It is an indication that all or almost all Fe^{3+} is located at one site, labeled *M*.

CHEMICAL DATA

Two chemical analyses of the type specimen were carried out with a CamScan MV2300 (Tescan Vega TS5130MM) digital scanning electron microscope equipped with YAG detectors of secondary and back-scattered electrons, a Link INCA energy-dispersion X-ray microanalysis instrument with a Si(Li) semiconductor detector (EDS mode, 15.7 kV, 1.5 nA, 8 μm beam diameter). Averaged analytical results are given in Table 2. We found Na, K, Sr, Ba, Pb, Zn, Ti, REE, S, Cl, and F to be below the detection limits, *i.e.*, <0.03–0.05 wt.%.

The amount of BeO was determined by inductively coupled plasma – atomic emission spectroscopy (ICP–AES). Analyses were performed on an ARL–3410 model with sequential spectrometer using solutions obtained by alkali fusion of two samples (0.00207 and 0.00691 g) mixed with 0.15000 g of lithium tetraborate and metaborate in the proportion 1:4 (eutectic mixture). The mean value (the average of two measurements, 8.23 and 8.58 wt.% BeO, respectively) is 8.41 wt.%, which corresponds to 3.63 *apfu* Be compared to the ideal 4 *apfu*. The low value is probably due to an admixture with cyrilovite (as can be seen in Fig. 2). We consequently used the BeO amount calculated for 4.00 *apfu* Be in the chemical composition (Table 2).

The amount of H_2O was determined by the Penfield method (average of two measurements, 12.44 and 12.68 wt.% respectively). There is no evidence of CO_3^{2-} groups in IR data, and no gas evolution was observed with HCl treatment.

The empirical formula for the type specimen, calculated based on six (PO_4) groups per formula unit, is $\mathrm{Ca}_{1.89}(\square_{1.45}\mathrm{Mn}^{2+}_{0.55})_{\Sigma 2.00}(\mathrm{Fe}^{3+}_{1.69}\mathrm{Mg}_{0.86}\mathrm{Mn}^{2+}_{0.68}\mathrm{Fe}^{2+}_{0.59}\mathrm{Al}_{0.18})_{\Sigma 4.00}\mathrm{Be}_{4.00}(\mathrm{PO}_4)_6\,[(\mathrm{OH})_{4.75}(\mathrm{H_2O})_{1.25}]\bullet 3.90\mathrm{H_2O}$. The partially empty site (*D*) is populated by

TABLE 2. THE CHEMICAL COMPOSITION OF RUIFRANCOITE

	1	2	3	4	4 (range)	5	5 (range)	6	6 (range)	7	8	8 (range)	9
CaO wt%	9.72	9.90	9.81	7.47	(7.14-7.72)	9.75	(9.57-9.94)	9.25	(9.05-9.49)	7.68	9.39	(8.98-9.78)	9.77
K_2O				0.86	(0.70-0.96)						0.12	(0.00-0.36)	0.17
Na_2O											0.29	(0.00-0.87)	0.74
BaO				0.08	(0.00-0.40)								
BeO	9.17	9.36	9.26							12.71			9.17
Fe_2O_3	11.43	13.60	12.51	21.89*	(21.07-23.77)	17.23*	(15.54-17.94)	14.14*	(12.85-15.08)	13.49	18.93*	(15.60-25.21)	10.05
FeO	3.62	4.25	3.93							6.32			5.69
MgO	4.70	1.76	3.23	2.94	(2.49-3.10)	1.00	(0.47-1.84)	3.07	(1.63-5.28)	-	3.29	(1.66-4.95)	4.75
MnO	7.37	8.84	8.10	1.54	(1.29-2.06)	11.00	(10.42-11.90)	8.32	(4.20-11.26)	10.14	6.05	(3.12-10.48)	6.80
Al_2O_3	0.57	1.16	0.86	1.11	(0.54-2.26)	0.93	(0.59-1.27)	1.20	(0.55-1.66)		0.65	(0.28-1.35)	0.42
ZnO				0.51	(0.35-0.73)						1.06	(0.00-2.83)	0.08
P_2O_5	39.04	39.88	39.46	36.29	(35.08-37.06)	42.50	(42.20-42.81)	36.51	(35.81-37.29)	37.98	36.44	(35.61-38.98)	36.77
SiO_2				0.29	(0.23-0.32)			0.23	(0.09-0.48)		0.33	(0.00-1.81)	0.83
H_2O	12.56	12.56	12.56							11.68			11.85
Total	98.18	101.31	99.72							100.00			97.09
Ca *apfu*	1.89	1.89	1.89	1.55		1.74		1.91		1.54	1.94		1.96
K				0.21							0.03		0.04
Na											0.11		0.27
Ba				0.01									
Be	4	4	4										4.13
Fe^{3+}	1.56	1.82	1.69	3.18*		2.16*		2.05*		1.90	2.74*		1.42
Fe^{2+}	0.55	0.63	0.59							0.99			0.89
Mg	1.27	0.47	0.86	0.85		0.25		0.88		-	0.94		1.33
Mn	1.13	1.33	1.23	0.25		1.55		1.36		1.60	0.99		1.08
Al	0.12	0.24	0.18	0.25		0.18		0.27			0.15		0.09
Zn				0.07							0.15		0.01
ΣM	4.63	4.49	4.55	4.60		4.14		4.56		4.49	4.97		4.82
P	6	6	6	5.94		6		5.96		6	5.94		5.84
Si				0.06				0.04			0.06		0.16
OH	4.72	4.82	4.75										6.09
H_2O	5.25	5.03	5.14										4.36
OH +H_2O	9.97	9.85	9.89										10.45

Columns 1 to 7. Sapucaia pegmatite, Galiléia, Minas Gerais. 1 and 2. Type specimen of ruifrancoite. Electron-microprobe data. Standards employed: wollastonite (Ca), diopside (Mg), $MnTiO_3$ (Mn), Fe (Fe), Al_2O_3 (Al), $LaPO_4$ (P). 3. Mean of columns 1 and 2. 4. Mn-poor ruifrancoite. Mean result of five energy-dispersion analyses. Standards employed: MgO (Mg), Al_2O_3 (Al), quartz (Si), GaP (P), MAD (K), wollastonite (Ca), Mn (Mn), Fe (Fe), Zn (Zn), BaF_2 (Ba). 5. Mn-rich ruifrancoite. Mean result of five energy-dispersion analyses. Standards employed: as for 4. 6. Mn-rich ruifrancoite. Mean result of eight electron-microprobe analyses. Standards employed: wollastonite (Ca,Si), diopside (Mg), olivine (Mn,Fe), anorthite (Al), chlorapatite (P). 7. Ruifrancoite studied by Lindberg (1958), considered as "roscherite". Results of wet-chemical analyses. Columns 8 and 9. Lavra Pomarolli, Linópolis, Divino das Laranjeiras, Minas Gerais. 8 from Atencio *et al.* (2005), mean result of seven energy-dispersion analyses. 9. Results of wet-chemical analyses. * Fe total. ΣM: sum of octahedrally coordinated cations.

larger cations (see below). For this reason, we contend that *D* contains Mn^{2+}. The simplified formula is $Ca_2(\square,Mn^{2+})_2(Fe^{3+},Mg,Mn^{2+},Fe^{2+},Al)_4Be_4(PO_4)_6(OH)_4(OH,H_2O)_2\bullet 4H_2O$. The ideal end-member formula of ruifrancoite is $Ca_2\square_2Fe^{3+}{}_4Be_4(PO_4)_6(OH)_6\bullet 4H_2O$. Partial chemical data for other samples of ruifrancoite are also included in Table 2 in order to show the chemical variability of the mineral.

CRYSTALLOGRAPHY

Single-crystal electron-microdiffraction patterns for ruifrancoite were obtained by transmission electron microscopy using a Tesla BS–540 instrument. We used an accelerating voltage of 80 kV. The samples were prepared by the suspension method. The data revealed that the mineral is monoclinic, space group *C*2/*c*, *a* 15.93(1), *b* 11.86(1), *c* 6.635(5) Å, β 96(1)°, *V* 1247 Å^3, *Z* = 2. X-ray powder-diffraction data (obtained with a Siemens D5000 diffractometer equipped with a Göbel mirror and a position-sensitive detector, using Cu*K*α radiation and 40 kV and 40 mA) are presented in Table 3. Unit-cell parameters refined from powder data are *a* 15.911(7), *b* 11.894(7), *c* 6.625(7) Å, β 94.5(1)°, *V* 1250(1) Å^3. The *a*:*b*:*c* proportions are: 1.3377:1:0.5570. The compatibility index $1 - (K_P/K_C)$ is –0.015, with measured density, and –0.023, with calculated density, indicating, according to Mandarino (1979), superior and excellent compatibility, respectively.

TABLE 3. X-RAY POWDER-DIFFRACTION DATA FOR RUIFRANCOITE

I (%)	d_{meas} (Å)	d_{calc} (Å)	*h*	*k*	*l*	I (%)	d_{meas} (Å)	d_{calc} (Å)	*h*	*k*	*l*
44	9.485	9.516	$\bar{1}$	1	0	22	3.065	3.065	5	1	0
18	7.924	7.931	2	0	0	11	2.974	2.973	0	4	0
100	5.943	5.947	0	2	0	17	2.819	2.823	$\bar{3}$	1	2
9	5.515	5.550	$\bar{1}$	1	1	41	2.784	2.784	2	4	0
65	4.821	4.831	3	1	0	42	2.643	2.644	6	0	0
7	4.399	4.419	0	2	1	6	2.539	2.540	2	4	1
8	3.958	3.950	$\bar{2}$	2	1	13	2.414	2.414	$\bar{4}$	2	2
18	3.348	3.352	$\bar{1}$	3	1	11	2.225	2.226	7	1	0
14	3.299	3.296	1	3	1	9	2.035	2.035	$\bar{2}$	2	3
44	3.176	3.172	3	3	0	12	1.982	1.985	0	6	0
25	3.146	3.137	$\bar{2}$	0	2	11	1.650	1.650	8	4	0

CRYSTAL CHEMISTRY OF THE ROSCHERITE GROUP

The roscherite-group minerals can be subdivided into two subgroups according to symmetry (Tables 4, 5). Most of the investigated species of this group (roscherite, zanazziite, greifensteinite, guimarãesite, ruifrancoite) are monoclinic, space group *C*2/*c* (Fanfani *et al.* 1975, Chukanov *et al.* 2002, 2007, Rastsvetaeva *et al.* 2002, 2004a, 2005, Barinova *et al.* 2004, Atencio *et al.* 2005). Other roscherite-group minerals (atencioite, footemineite) are triclinic, space group $P\bar{1}$ (Fanfani *et al.* 1977, Leavens *et al.* 1990, Taucher *et al.* 1992,

TABLE 4. CRYSTAL-CHEMICAL DATA FOR MONOCLINIC (*C*2/*c*) ROSCHERITE-GROUP MINERALS

	Formula		Mean cation–anion distances, Å		*D*-site occupancy
	D site $\Sigma = 2.00$	*M* site $\Sigma = 4.00$	*D* site	*M* site	
Roscherite[1]	$\square_{1.2}Mn_{0.6}Mg_{0.2}$	$Mn_{2.2}Fe_{1.6}Al_{0.2}$	2.175	2.145	40%
Zanazziite[2,3]	$\square_{0.99}Mg_{0.61}Fe_{0.40}$	$Mg_{2.38}Fe^{2+}{}_{1.08}Al_{0.31}Mn_{0.23}Fe^{3+}{}_{0.10}$	2.179	2.096	50.5%
Greifensteinite[4]	$\square_{1.34}Mn_{0.66}$	$Fe^{2+}{}_{3.46}Mn_{0.22}Al_{0.22}Mg_{0.10}$	2.171	2.132	33%
Zn-bearing greifensteinite[5]	$\square_{1.32}Mn_{0.68}$	$Fe^{2+}{}_{2.35}Zn_{0.72}Mn_{0.46}Al_{0.28}Mg_{0.19}$	2.196	2.106	34%
Guimarãesite[6]	$\square_{1.16}Zn_{0.50}Mn_{0.34}$	$Zn_{2.11}Mg_{1.11}Fe^{2+}{}_{0.41}Al_{0.37}$			42%
Ruifrancoite[7]	$\square_{1.45}Mn^{2+}{}_{0.55}$	$Fe^{3+}{}_{1.69}Mg_{0.86}Mn_{0.68}Fe^{2+}{}_{0.59}Al_{0.18}$			27.5%

[1] Roscherite (Rastsvetaeva *et al.* 2005, modified): $Ca_2Mn^{2+}{}_5Be_4(PO_4)_6(OH)_4\bullet 6H_2O$;
a 15.936, *b* 11.964, *c* 6.665 Å, β 94.78º.
[2, 3] Zanazziite (Fanfani *et al.* 1975, Leavens *et al.* 1990): $Ca_2Mg_5Be_4(PO_4)_6(OH)_4\bullet 6H_2O$;
a 15.874, *b* 11.854, *c* 6.605 Å, β 95.35°.
[4] Greifensteinite (Rastsvetaeva *et al.* 2002): $Ca_2Fe^{2+}{}_5Be_4(PO_4)_6(OH)_4\bullet 6H_2O$;
a 15.903, *b* 11.885, *c* 6.677 Å, β 94.68°.
[5] Zn-bearing greifensteinite (Barinova *et al.* 2004):
a 15.941, *b* 11.877, *c* 6.625 Å, β 95.09°.
[6] Guimarãesite (Chukanov *et al.* 2007): $Ca_2Zn_5Be_4(PO_4)_6(OH)_4\bullet 6H_2O$;
a 15.98, *b* 11.84, *c* 6.63 Å, β 95.15°.
[7] Ruifrancoite (this paper): $Ca_2Fe^{3+}{}_4Be_4(PO_4)_6(OH)_6\bullet 4H_2O$;
a 15.92, *b* 11.91, *c* 6.61 Å, β 96.4º.

Rastsvetaeva *et al.* 2004b, Chukanov *et al.* 2006, Atencio *et al.* 2008).

Roscherite-group minerals have a three-dimensional heteropolyhedral framework formed by chains of corner-sharing PO_4 and BeO_4 tetrahedra extending in the [101] direction, which are linked by edge-sharing chains of MO_6 octahedra (M stands for Mg, Mn, Fe, Al, or Zn) parallel to [110] and [$1\bar{1}0$] (Fig. 5). Calcium cations (with a seven-fold coordination) and H_2O molecules occupy cavities within the framework. The H_2O molecules are close to the free vertices of Ca polyhedra. The general crystallochemical formula of

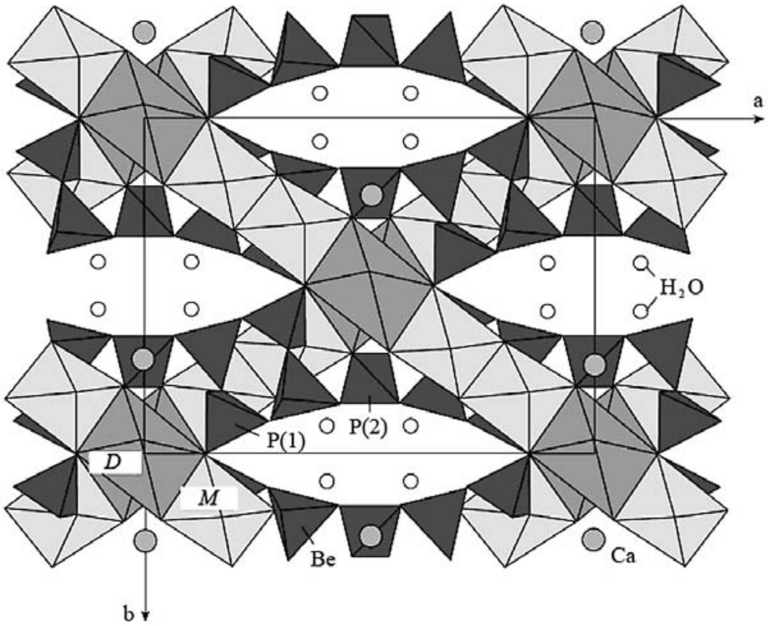

FIG. 5. The crystal structure of a monoclinic roscherite-group mineral.

TABLE 5. CRYSTAL-CHEMICAL DATA FOR TRICLINIC ($P\bar{1}$) ROSCHERITE-GROUP MINERALS

	Formula				Mean cation–anion distances, Å				*D*-site occupancy	
	D(1) site $\Sigma = 1.00$	*D*(2) site $\Sigma = 1.00$	*M*(1) site $\Sigma = 2.00$	*M*(2) site $\Sigma = 2.00$	*D*(1)	*D*(2)	*M*(1)	*M*(2)	*D*(1)	*D*(2)
Atencioite[1]	$Fe_{0.5}{}^{2+}Mn_{0.2}$ $Al_{0.1}\square_{0.2}$	$\square_{0.8}Mg_{0.2}$	$Mg_{1.1}Fe^{3+}{}_{0.5}$ $Fe^{2+}{}_{0.4}$	$Fe^{2+}{}_{1.1}Mg_{0.9}$	2.183	2.132	2.106	2.122	80%	20%
Footemineite[2]	$Mn^{2+}{}_{0.90}$ $\square_{0.10}$	$\square_{0.78}Li_{0.17}$ $Mg_{0.05}$	$Mn^{2+}{}_{1.63}$ $Fe^{2+}{}_{0.22}$ $\square_{0.08}Fe^{3+}{}_{0.05}$ $Al_{0.02}$	$Mn^{2+}{}_{1.63}$ $Fe^{2+}{}_{0.22}$ $\square_{0.08}Fe^{3+}{}_{0.05}$ $Al_{0.02}$	2.23	2.17	2.19	2.19	90%	22%

[1] Atencioite (Rastsvetaeva *et al.* 2004b): $Ca_2Fe^{2+}Mg_2Fe^{2+}{}_2Be_4(PO_4)_6(OH)_4{\bullet}6H_2O$; *a* 6.668(1), *b* 9.879(2), *c* 9.883(1) Å, α 73.53(1), β 85.60(1), γ 86.93(1)°.
[2] Footemineite (Atencio *et al.* 2008): $Ca_2Mn^{2+}Mn^{2+}{}_2Mn^{2+}{}_2Be_4(PO_4)_6(OH)_4{\bullet}6H_2O$; *a* 6.788(2), *b* 9.972(3), *c* 10.014(2) Å, α 73.84(2), β 85.34(2), γ 87.44(2)°.

monoclinic roscherite-group minerals can be written as $Ca_2D_2M_4Be_4(PO_4)_6(OH)_4X_2{\bullet}4H_2O$, where D and M are octahedral sites occupied by Mg, Mn^{2+}, Fe^{2+}, Fe^{3+}, Zn, or Al. The D sites are largely vacant, their occupancy usually being 1/3 to 1/2 per formula unit, and X represents OH or H_2O.

As seen from Table 4, in all structurally investigated monoclinic roscherite-group minerals, average cation–anion distances at the D site are longer than those at the M site. This comparison shows that the D site is preferred by larger divalent cations than by smaller cations like Fe^{3+} or Al^{3+}. In the structural investigation of what became zanazziite (Fanfani *et al.* 1975), Al was assigned to the D site. But later, Fanfani *et al.* (1977) stated that "considerations on the atomic arrangement do not exclude the possibility of roscherites where the octahedral trivalent sites are partially or completely occupied by divalent cations". Moreover, as was shown later by Leavens *et al.* (1990), the sample investigated contains <1/3 *apfu* Al. Taking into account the smaller size of Fe^{3+} and Al^{3+} over Mg^{2+}, they placed those cations at the M site, whereas for the D site they gave the composition $\square_{0.99}Mg_{0.61}Fe_{0.40}$.

In the structurally investigated monoclinic roscherite-group minerals (roscherite, zanazziite and greifensteinite), divalent cations prevail over trivalent cations at the D site. By analogy with earlier data by Fanfani *et al.* (1975), Rastsvetaeva *et al.* (2005) assigned 0.46 Al to the D site of roscherite. As noted above, this seems incorrect, and we assign a more probable distribution of cations in Table 4. For ruifrancoite, the Mössbauer spectroscopy data also indicate that all Fe^{3+} is located at the M site. In accordance with single-crystal structural data for other monoclinic roscherite-group minerals, the D site is 50–67% vacant; in ruifrancoite, it is 72.5% vacant. Chukanov & Pekov (2005) demonstrated, by comparative analysis of multiple structures based on heteropolyhedral frameworks, that cations such as Al, Fe^{3+} or Mn^{3+} usually do not occupy sites tending to be vacant, whereas for sites occupied only by cations such Mg, Zn, Fe^{2+} or Mn^{2+}, vacancies are more frequent (Table 6). Consequently, Al and Fe^{3+} will not be expected at the D site.

The lowering of symmetry in the triclinic roscherite-group minerals (atencioite and footemineite) is related to the transformation of the D and M sites into pairs of non-equivalent sites occupied by different cations (Fig. 6). The general crystallochemical formula of triclinic roscherite-group minerals can be written as $Ca_2D(1)D(2)M(1)_2M(2)_2Be_4(PO_4)_6(OH)_4X_2{\bullet}4H_2O$. Both $D(1)$ and $D(2)$ sites preserve their tendency to be vacant; their total occupancy is <50%. However, the

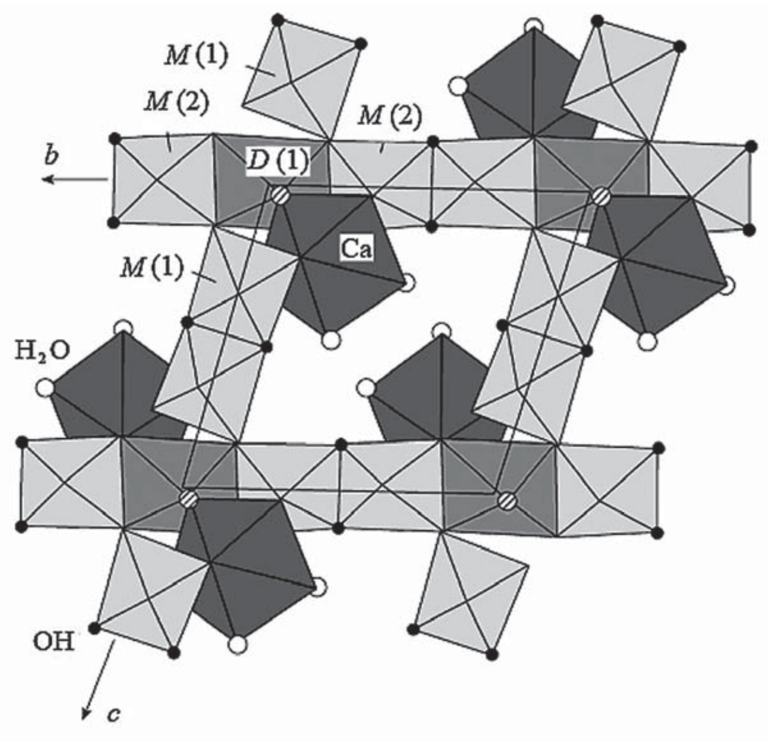

FIG. 6. The crystal structure of a triclinic roscherite-group mineral. The vacant $D(2)$ octahedron is not shown.

TABLE 6. PROPERTIES OF CATIONS IN MINERALS WITH HETEROPOLYHEDRAL FRAMEWORKS[§]

Cation site	CN	Ionic radius (Å)	Main cations	Tendency to			Stretching frequencies cm^{-1}
				substitution	vacancies	splitting	
T	4	< 0.5	Si, Al, P, B, Be*	–	–	–	850-1100
*M***	6	0.6-0.7	Ti, Nb, Zr, Fe^{3+}, Y, Mn^{3+}, Sn^{4+}, W^{6+}	+	–	–	550-750
*D****	6	0.7-1.0	Mn^{2+}, Fe^{2+}, Mg, Zn, Ca	+	+	–	400-500
A	> 6	1.1-1.5	Na, K, Ca, Sr, Ba, Pb^{2+}, $(H_3O)^+$	+	+	+	< 400

[§] After Chukanov & Pekov (2005). CN: coordination number.
* For Zn with 4-fold coordination, the effective ionic radius is ~0.6 Å.
** High field-strength octahedrally coordinated cations, with charges > 2.
*** Medium field-strength divalent octahedrally coordinated cations.

D(1) site is filled, and *D*(2) is empty in both atencioite and footemineite (Rastsvetaeva *et al.* 2004b, Atencio *et al.* 2008).

CONCLUDING REMARKS

Ruifrancoite, a Fe^{3+}-dominant roscherite-group mineral, is considered to have formed in an oxidizing environment. The presence of two other Fe^{3+}-rich phosphates, cyrilovite and meurigite, in close association with ruifrancoite, reinforces this conjecture. Another possible indicator of high activity of oxygen at the late stages of mineral formation in granitic pegmatites at the Sapucaia mine is the occurrence of a Fe^{3+}-rich lipscombite-type mineral described by Lindberg (1962). In other samples of Fe-rich roscherite-group minerals (greifensteinite and atencioite), Mössbauer spectra show strong predominance of Fe^{2+} over Fe^{3+}.

The "roscherite" previously described by Lindberg (1958) and Lindberg & Pecora (1958) at the Sapucaia mine, and the "Fe^{3+}-dominant species" described by Atencio *et al.* (2005) at Pomarolli farm, Linópolis, Divino das Laranjeiras County, Minas Gerais, Brazil, are also probably ruifrancoite.

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