

1 **REVISION 1**

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5 **Césarferreiraite, Fe<sup>2+</sup>Fe<sup>3+</sup><sub>2</sub>(AsO<sub>4</sub>)<sub>2</sub>(OH)<sub>2</sub>·8H<sub>2</sub>O, from Eduardo mine, Conselheiro Pena,**  
6 **Minas Gerais, Brazil: second arsenate in the laueite mineral group**

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9 <sup>1</sup>Ricardo Scholz, <sup>2</sup>Nikita V. Chukanov, <sup>3</sup>Luiz A.D. Menezes Filho, <sup>4</sup>Daniel Atencio,  
10 <sup>1</sup>Leonardo Lagoeiro, <sup>5</sup>Fernanda M. Belotti, <sup>3</sup>Mário L.S.C. Chaves, <sup>3</sup>Antônio W. Romano,  
11 <sup>6</sup>Paulo R. Brandão, <sup>7</sup>Dmitriy I. Belakovskiy and <sup>8</sup>Igor Pekov

12  
13 <sup>1</sup>Universidade Federal de Ouro Preto (UFOP), Escola de Minas, Departamento de Geologia,  
14 Campus Morro do Cruzeiro, 35400-000, Ouro Preto, Minas Gerais, Brazil; e-mail:  
15 [r\\_scholz\\_br@yahoo.com](mailto:r_scholz_br@yahoo.com)

16 <sup>2</sup>Institute of Problems of Chemical Physics, Russian Academy of Sciences, Chernogolovka,  
17 Moscow region, 142432 Russia

18 <sup>3</sup>Instituto de Geociências, Universidade Federal de Minas Gerais, Av. Antônio Carlos, 6627,  
19 31270-901, Belo Horizonte, Minas Gerais, Brazil

20 <sup>4</sup>Instituto de Geociências, Universidade de São Paulo, Rua do Lago, 562, 05508-080, São  
21 Paulo, SP, Brazil

22 <sup>5</sup>Universidade Federal de Itajubá (UNIFEI), Campus Itabira, Itabira, Minas Gerais, Brazil

23 <sup>6</sup>Escola de Engenharia, Universidade Federal de Minas Gerais, Av. Antônio Carlos, 6627,  
24 31270-901, Belo Horizonte, Minas Gerais, Brazil

25 <sup>7</sup>Fersman Mineralogical Museum of the Russian Academy of Sciences, Leninsky Prospekt 8-  
26 2, 117071 Moscow, Russia

27 <sup>8</sup>Faculty of Geology, Moscow State University, Vorobiev Gory, 119899 Moscow, Russia

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29 **ABSTRACT**

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31 Césarferreiraite,  $\text{Fe}^{2+}\text{Fe}^{3+}_2(\text{AsO}_4)_2(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ , is a new laueite-group mineral (IMA  
32 2012-099) of triclinic symmetry, from Eduardo pegmatite mine, Conselheiro Pena  
33 municipality, Minas Gerais, Brazil. Intimately associated minerals are pharmacosiderite,  
34 scorodite and earlier arsenopyrite, and probably césarferreiraite replaces the latter. It occurs as  
35 fibrous to tabular aggregates up to 2 mm. Single crystals, up to 10  $\mu\text{m}$  long with a thickness of  
36 about 1-2  $\mu\text{m}$ , are elongated along [001] and flattened on (100). The fibers have almost  
37 rectangular cross-section apparently bound by the {100} and {010} pinacoid forms. Colour  
38 and streak are pale to greenish yellow. Luster is vitreous; individual crystals are transparent  
39 and masses are translucent. Cleavage is distinct, presumably on {010} and {100}. Calculated  
40 density is 2.934  $\text{g cm}^{-3}$ . The mineral is biaxial (+),  $n$  (min) = 1.747(3),  $n$  (max) = 1.754(3)  
41 (589 nm). IR spectrum of césarferreiraite is unique and can be used for the identification of  
42 the mineral. Chemical composition ( $n = 4$ , WDS, calculated for the condition  $\text{Fe}^{2+}:\text{Fe}^{3+} = 1:2$ ,  
43  $\text{H}_2\text{O}$  for the ideal structural formula, wt.%) is: FeO 11.50,  $\text{Fe}_2\text{O}_3$  25.56, CaO 15.41,  $\text{As}_2\text{O}_5$   
44 33.51,  $\text{H}_2\text{O}$  26.01, total 100.12. The empirical formula (based on 18 O *apfu*) is  
45  $\text{Fe}^{2+}_{0.98}\text{Fe}^{3+}_{1.96}[(\text{AsO}_4)_{1.79}(\text{PO}_4)_{0.31}](\text{OH})_{1.52} \cdot 8.08\text{H}_2\text{O}$ . The strongest eight X-ray powder-  
46 diffraction lines [ $d$  in  $\text{\AA}(I)(hkl)$ ] are: 9.85(95)(010), 6.35(100)(001), 3.671(29)(-121),  
47 3.158(32)(1-30), 2.960(39)(02-2), 2.884(35)(-131), 2.680(29)(-211), and 2.540(23)(-210).  
48 Unit-cell parameters refined from powder data indexed by analogy with related laueite-group  
49 minerals (space group:  $P\bar{1}$ ) are:  $a = 5.383(2)$ ,  $b = 10.363(3)$ ,  $c = 6.878(2)$   $\text{\AA}$ ,  $\alpha = 96.42(4)$ ,  $\beta =$   
50  $109.19(3)$ ,  $\gamma = 102.30(2)^\circ$ ,  $V = 347.1(2)$   $\text{\AA}^3$ , and  $Z = 1$ . Gladstone-Dale compatibility is -0.020  
51 (excellent). Césarferreiraite is the arsenate analogue of ferrolaueite.

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53 **Keywords:** césarferreiraite, new mineral, granitic pegmatite, Conselheiro Pena, Minas Gerais,  
54 Brazil, laueite group, iron arsenate.

55

## 56 INTRODUCTION

57 In May 2009, during the preparation of the Eastern Brazilian Pegmatite field trip for  
58 the 4<sup>th</sup> International Symposium on Granitic Pegmatites (PEG 2009), two of the authors (R.S.  
59 and A.W.R.) found a centimetric cavity covered by pharmacosiderite and an unusual  
60 yellowish fibrous aggregate. The latter turned out a new mineral species belonging to the  
61 laueite group, with the formula  $\text{Fe}^{2+}\text{Fe}^{3+}_2(\text{AsO}_4)_2(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ . It was approved by the IMA  
62 CNMNC on March 1, 2013 with the name césarferreiraite (IMA 2012-099).

63 The name is in honour of Professor César Mendonça Ferreira (b. 1942). Graduating as  
64 a Geology Engineer in the School of Mines of Ouro Preto in 1970, Professor Ferreira  
65 developed a long career as Professor in Mineralogy and Gemology. He engaged in many  
66 studies of the mineralogy of ores and in the field of geometallurgy. During the last 15 years he  
67 was responsible for establishing the Gemological Laboratory of the Federal University of  
68 Ouro Preto. Professor Ferreira has agreed to the naming of the mineral. The holotype has been  
69 deposited in the mineralogical collection of the Museu de Ciência e Técnica, Escola de Minas,  
70 Universidade Federal de Ouro Preto, Praça Tiradentes, Centro, 35400-000 – Ouro Preto,  
71 Minas Gerais, Brazil, registration number SAA-011.

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## 73 GEOLOGICAL SETTING AND OCCURRENCE

74 Césarferreiraite occurs in a 1 cm<sup>3</sup> miarolitic cavity in albite in the Eduardo pegmatite  
75 mine (“Lavra do Eduardo”, in Portuguese), near Boa Vista creek, Conselheiro Pena  
76 municipality, Minas Gerais, Brazil (19°4'53.09"S 41°30'34.10"W), in association with  
77 pharmacosiderite, scorodite and earlier arsenopyrite and probably replaces the latter.

78 Césarferreiraite is known in only one block of pegmatite rock, and total amount of available  
79 substance did not exceed 2 mg.

80 The Eduardo mine (erroneously quoted as Boa Vista mine, another famous mine, by  
81 Bermanec *et al.*, 2011) is located in the Conselheiro Pena pegmatite district (Figure 1), one of  
82 the subdivisions of the Eastern Brazilian Pegmatite province that encompasses an area of  
83 about 150,000 km<sup>2</sup>, extending from Bahia to Rio de Janeiro states (Pedrosa *et al.* 2011).  
84 Around 90% of the province is situated in the eastern part of the State of Minas Gerais. The  
85 Conselheiro Pena pegmatite district covers an area of about 5,000 km<sup>2</sup>, in the middle part of  
86 the Doce River basin, approximately 360 km at NE of the city of Belo Horizonte

87 The Conselheiro Pena pegmatite district is inserted in the central domain of the  
88 Araçuaí mobile belt (Almeida 1977), formed during the Brasiliano orogeny (630-490 Ma) by  
89 accretion to the eastern margin of the São Francisco craton (Pedrosa Soares *et al.* 2011). In  
90 this area, several suites of granitoid rocks are distinguished (*e.g.* Urucum and Palmital of  
91 Eocambrian to Paleozoic age, and Galileia of Neoproterozoic age), intruding schists of the  
92 Neoproterozoic São Tomé formation (Nalini *et al.* 2008).

93 The Eduardo pegmatite was mined for industrial feldspar and with minor importance  
94 gemstones and samples for the collectors market, mainly phosphates (red variscite,  
95 phosphosiderite and cyrilovite) and arsenates (scorodite and pharmacosiderite). At present,  
96 the Eduardo pegmatite is mined out. The pegmatite is heterogeneous with well-developed  
97 mineralogical and textural zoning. It has symmetric lens shape with the longer axis trending  
98 NW-SE and steeply dipping. The minimum extension is around 20 m, and the thickness is 12  
99 m. The pegmatite is hosted by quartz-mica schist with garnet, staurolite and sillimanite of the  
100 São Tomé Formation. Tourmalinization is observed in the contact between the pegmatite and  
101 the host rock. Hydrothermal and metasomatic fluids were responsible for the albitization and

102 development of miarolitic cavities, and a complex secondary phosphate assemblage was  
103 described by Chaves *et al.* (2005).

104 The pegmatite crops out on a steep hillside, and, at the uppermost end, pinches out to  
105 less than 1 m in width. Other minerals found in the pegmatite, including secondary species,  
106 are albite, garnets of the almandine-spessartine solid-solution series, arenopyrite, beryl,  
107 bismuth, bütschliite, cryptomelane, cyrilovite, löllingite, fourmarierite, frondelite-  
108 rockbridgeite solid-solution series, heterosite, hureaulite, kaolinite, leucophosphate,  
109 manganese oxides, metatorbenite, microcline, muscovite, pharmacosiderite, phosphosiderite,  
110 quartz, saleeite, schorl, scorodite, spodumene, strengite, triphylite, ushkovite and variscite  
111 (Bermanec *et al.* 2011).

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### 113 **APPEARANCE AND PHYSICAL PROPERTIES**

114 Césarferreiraite occurs as fibrous (random and subparallel) to tabular aggregates up to  
115 2 mm. Single crystals are imperfect, flattened and lath-shaped. Crystals, up to 10  $\mu\text{m}$  long  
116 with a thickness of about 1-2  $\mu\text{m}$ , are elongated along [001] and flattened on (100). The fibers  
117 have a near-rectangular cross-section apparently bound by the {100} and {010} pinacoid  
118 forms (Figures 2 and 3). Twinning was not observed. The color and also the streak of  
119 césarferreiraite vary from pale yellow to greenish yellow. The luster is vitreous; the individual  
120 crystals are transparent and masses are translucent. The mineral is non-fluorescent under  
121 short- and long-wave ultraviolet light. Due to the small sizes of crystals and too little pure  
122 material, the Mohs hardness was not measured, but the mineral is easily crushed between two  
123 glass slides. The tenacity is brittle and the cleavage is distinct, presumably on {010} and  
124 {100}. Parting was not observed and fracture is uneven. Density could not be measured  
125 because of small crystal sizes. Calculated density based on the empirical formula is 2.934 g  
126  $\text{cm}^{-3}$ .

127 Acicular crystals of *césarferreiraite* consist of very thin fibrous differently oriented  
128 blocks. Therefore only minimum and maximum refractive indices were measured. The  
129 mineral is biaxial (+),  $n$  (min) = 1.747(3),  $n$  (max) = 1.754(3) (589 nm). The mean  $n$  value is  
130 about 1.751.

131

### 132 CHEMICAL COMPOSITION

133 Chemical analyses ( $n = 4$ ) were carried out using a Jeol JXA8900R electron  
134 microprobe housed in the Physics Department of the Federal University of Minas Gerais,  
135 Belo Horizonte (WDS mode, 15 kV, 20 nA, *ca* 5  $\mu\text{m}$  beam diameter).  $\text{CO}_2$  was not analysed  
136 because bands for  $\text{CO}_3^{2-}$  anions are absent in the IR spectrum. Contents of Mg, Al and Mn are  
137 below detection limits. Too little material was available for a direct determination of  $\text{H}_2\text{O}$ , but  
138 its presence was confirmed by IR spectroscopy. Analytical data are given in Table 1.

139 The empirical formula (based on 18 O *apfu*) is, with rounding errors,  
140  $\text{Fe}^{2+}_{0.98}\text{Fe}^{3+}_{1.96}[(\text{AsO}_4)_{1.79}(\text{PO}_4)_{0.31}](\text{H}_{17.68}\text{O}_{9.60})$ , or  $\text{Fe}^{2+}_{0.98}\text{Fe}^{3+}_{1.96}[(\text{AsO}_4)_{1.79}(\text{PO}_4)_{0.31}](\text{OH})_{1.52}\cdot$   
141  $8.08\text{H}_2\text{O}$ . The simplified formula is  $\text{Fe}^{2+}\text{Fe}^{3+}_2(\text{AsO}_4)_2(\text{OH})_2\cdot 8\text{H}_2\text{O}$ , which requires FeO 11.52,  
142  $\text{Fe}_2\text{O}_3$  25.61,  $\text{As}_2\text{O}_5$  36.86,  $\text{H}_2\text{O}$  26.01, total 100.00 wt.%.

143 The IR spectrum of *césarferreiraite* (Figure 4) is unique and can be used for the  
144 identification of the mineral. Absorption bands and their assignments ( $\text{cm}^{-1}$ ; s – strong band, w  
145 – weak band, sh – shoulder) are 3456s, 3352s, 3200sh (O-H stretching vibrations of  $\text{H}_2\text{O}$   
146 molecules and OH groups), 1647, 1550w (bending vibrations of  $\text{H}_2\text{O}$  molecules), 1096sh, 1050,  
147 1026 (asymmetric stretching vibrations of  $\text{PO}_4^{3-}$  anions), 860sh, 830s, 759s (asymmetric  
148 stretching vibrations of  $\text{AsO}_4^{3-}$  anions), 592w (bending vibrations of  $\text{PO}_4^{3-}$  anions), 470s (lattice  
149 mode involving  $\text{Fe}^{3+}$ -O and  $\text{Fe}^{2+}$ -O stretching and possibly low-frequency vibrations of anions  
150 and water molecules). The presence of two bands in the range 1500-1700  $\text{cm}^{-1}$  indicates the

151 presence of non-equivalent H<sub>2</sub>O molecules in the mineral. Bands characteristic of CO<sub>3</sub><sup>2-</sup> anions  
152 and B-O bonds are absent in the IR spectrum.

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## 154 **CRYSTALLOGRAPHY**

155 Powder X-ray diffraction data were obtained with a D8 Advance DaVinci  
156 diffractometer using CuK $\alpha$  radiation (Table 2). Unit-cell parameters refined (CellCalc  
157 program, Miura 2003) from powder data indexed by analogy with related laueite-group  
158 minerals (triclinic, space group:  $P\bar{1}$ ) are as follows:  $a = 5.383(2)$ ,  $b = 10.363(3)$ ,  $c = 6.878(2)$   
159  $\text{\AA}$ ,  $\alpha = 96.42(4)$ ,  $\beta = 109.19(3)$ ,  $\gamma = 102.30(2)^\circ$ ,  $V = 347.1(2) \text{\AA}^3$ , and  $Z = 1$ . The  $a:b:c$  ratio  
160 calculated from the unit cell parameters is 0.783:1:1.507. Gladstone-Dale compatibility 1 –  
161  $(K_p/K_c) = -0.020$  (excellent) using the empirical formula and the average refractive index. Due  
162 to the tiny size of single crystals forming friable aggregates, single-crystal diffraction data  
163 were not obtained. However, the analogy with laueite-group minerals in stoichiometry and X-  
164 ray powder diffraction data, as well as the excellent compatibility confirms the correctness of  
165 the data obtained.

166 The laueite group (Krivovichev 2004) includes seven phosphates (ferrolaueite,  
167 gordonite, laueite, mangangordonite, paravauxite, sigloite, ushkovite) and only two arsenates:  
168 maghrebite, recently described by Meisser *et al.* (2012), and césarferreiraite, here described.  
169 Stewartite and pseudolaueite are polymorphs of laueite. Kastningite is isostructural with  
170 stewartite and dimorph with mangangordonite. Maghrebite is the arsenate analogue of  
171 gordonite, and césarferreiraite is the arsenate analogue of ferrolaueite, recently published  
172 (Segeler *et al.* 2012; Ferraiolo 2012). Arsenate members of the laueite group substantially  
173 differ from its phosphate members in the values of unit-cell parameters  $\alpha$  and  $\gamma$ . The  
174 properties of césarferreiraite are compared with those of maghrebite and ferrolaueite in Table  
175 3.

176 **IMPLICATIONS**

177 The general formula of laueite-group minerals is  $M1M2_2(TO_4)_2(OH)_{2-3} \cdot 8-7H_2O$  where  
178  $M1$  is typically a bivalent cation,  $M2 = Fe^{3+}$  or Al,  $T = P$  or As. Minerals with  $M1 = Fe^{2+}$  can  
179 oxidize and transform to their  $Fe^{3+}$ -analogues. This phenomenon is typical for ferrous  
180 phosphates; in particular, sigloite forms pseudomorphs after paravauxite (compare with the  
181 transformations vivianite  $\rightarrow$  metavivianite, ferrostrunzite  $\rightarrow$  ferristrunzite). Bivalent state of  
182 iron in  $M1$  of Césarferreiraite is confirmed by excellent Gladstone-Dale compatibility.  
183 Césarferreiraite is the second arsenate belonging to the laueite group. The discovery of this  
184 additional arsenate suggests that there are several other (at least six) new arsenates to be  
185 discovered, taking into account only the cations that have been found in the phosphates  
186 described till now (see Table 4).

187

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Table 1. Analytical data for césarferreiraité.

<i>Constituent</i>	<i>wt%</i>	<i>Range</i>	<i>Standard Deviation</i>	<i>Probe standard</i>
FeO*	11.50	11.29-11.70	0.10	Magnetite
Fe <sub>2</sub> O <sub>3</sub> *	25.56	22.58-23.39	0.37	Magnetite
As <sub>2</sub> O <sub>5</sub>	33.51	30.80-36.96	2.57	FeAs <sub>2</sub>
P <sub>2</sub> O <sub>5</sub>	3.54	2.36-4.98	0.88	Astimex apatite
H <sub>2</sub> O**	26.01			
Total	100.12			

231 \*Calculated from the condition Fe<sup>2+</sup>:Fe<sup>3+</sup> = 1:2, taking into account that for laueite-group232 minerals M<sup>2+</sup>:M<sup>3+</sup> = 1:2. \*\*Calculated by stoichiometry for the ideal structural formula.

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Table 2. X-ray powder diffraction data for césarferreiraite.

$I_{\text{rel}}$	$d_{\text{obs}}, \text{Å}$	$d_{\text{calc}}, \text{Å}$	$h$	$k$	$l$
<b>95</b>	<b>9.85</b>	9.927	0	1	0
<b>100</b>	<b>6.35</b>	6.369	0	0	1
21	4.963	4.963	0	2	0
17	4.534	4.542	-1	1	1
13	3.997	3.993	1	1	0
<b>29</b>	<b>3.671</b>	3.671	-1	2	1
<b>32</b>	<b>3.158</b>	3.160	1	-3	0
<b>39</b>	<b>2.960</b>	2.958	0	2	-2
<b>35</b>	<b>2.884</b>	2.884	-1	3	1
<b>29</b>	<b>2.680</b>	2.680	-2	1	1
<b>23</b>	<b>2.540</b>	2.541	-2	1	0
8	2.475	2.474	2	-2	0
8	1.9968	1.9970	0	1	3
8	1.9968	1.9964	2	2	0
5	1.9129	1.9132	-1	5	1
7	1.8798	1.8800	2	3	-1
7	1.6252	1.6249	-2	5	2
7	1.6153	1.6153	-3	1	3

236 Table 3. Comparative data for Césarferreiraite and related minerals.

Mineral	Césarferreiraite	Maghrebite	Ferrolaueite
Simplified formula	$\text{Fe}^{2+}\text{Fe}^{3+}_2(\text{AsO}_4)_2$ (OH) <sub>2</sub> ·8H <sub>2</sub> O	$\text{MgAl}_2(\text{AsO}_4)_2$ (OH) <sub>2</sub> ·8H <sub>2</sub> O	$\text{Fe}^{2+}\text{Fe}^{3+}_2(\text{PO}_4)_2$ (OH) <sub>2</sub> ·8H <sub>2</sub> O
Symmetry	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
<i>a</i> , Å	5.383(2)	5.436(2)	5.34(8)
<i>b</i> , Å	10.363(3)	10.500(3)	10.63(8)
<i>c</i> , Å	6.878(2)	7.075(2)	7.21(8)
$\alpha$ , °	96.42(4)	97.701(7)	107.35(8)
$\beta$ , °	109.19(3)	110.295(5)	111.26(8)
$\gamma$ , °	102.30(2)	102.021(6)	71.27(8)
<i>Z</i>	1	1	1
Strong lines of the X-ray powder-diffraction pattern:	9.851 (95) 6.346 (100) 3.671 (29) 3.158 (32) 2.960 (39)	9.9 (100) 6.4 (90) 4.90 (80) 3.198 (60) 2.885 (60)	9.87 (90) 6.56 (100) 4.90 (80) 3.97 (60) 3.92 (50)
<i>d</i> , Å ( <i>I</i> <sub>rel</sub> )	2.884 (35) 2.680 (29)	2.622 (60) 4.08 (50)	3.28 (100) 3.10 (50)
<b>Optical data:</b>	$n_{\min} = 1.747(3)$ $n_{\max} = 1.754(3)$	$\alpha = 1.562(2)$ $\beta = 1.574(2)$	$\alpha = 1.610(3)$ $\beta = 1.665(3)$

Optical sign, $2V, ^\circ$	+	$\gamma = 1.586(2)$ – 87	$\gamma = 1.692(3)$ – 68(2)
Density, $\text{g cm}^{-3}$	2.934 (calc.)	2.60 (meas.) 2.46 (calc.)	2.23 (meas.) 2.22 (calc.)
References	This work	Meisser <i>et al.</i> (2012)	Segeler <i>et al.</i> (2012)

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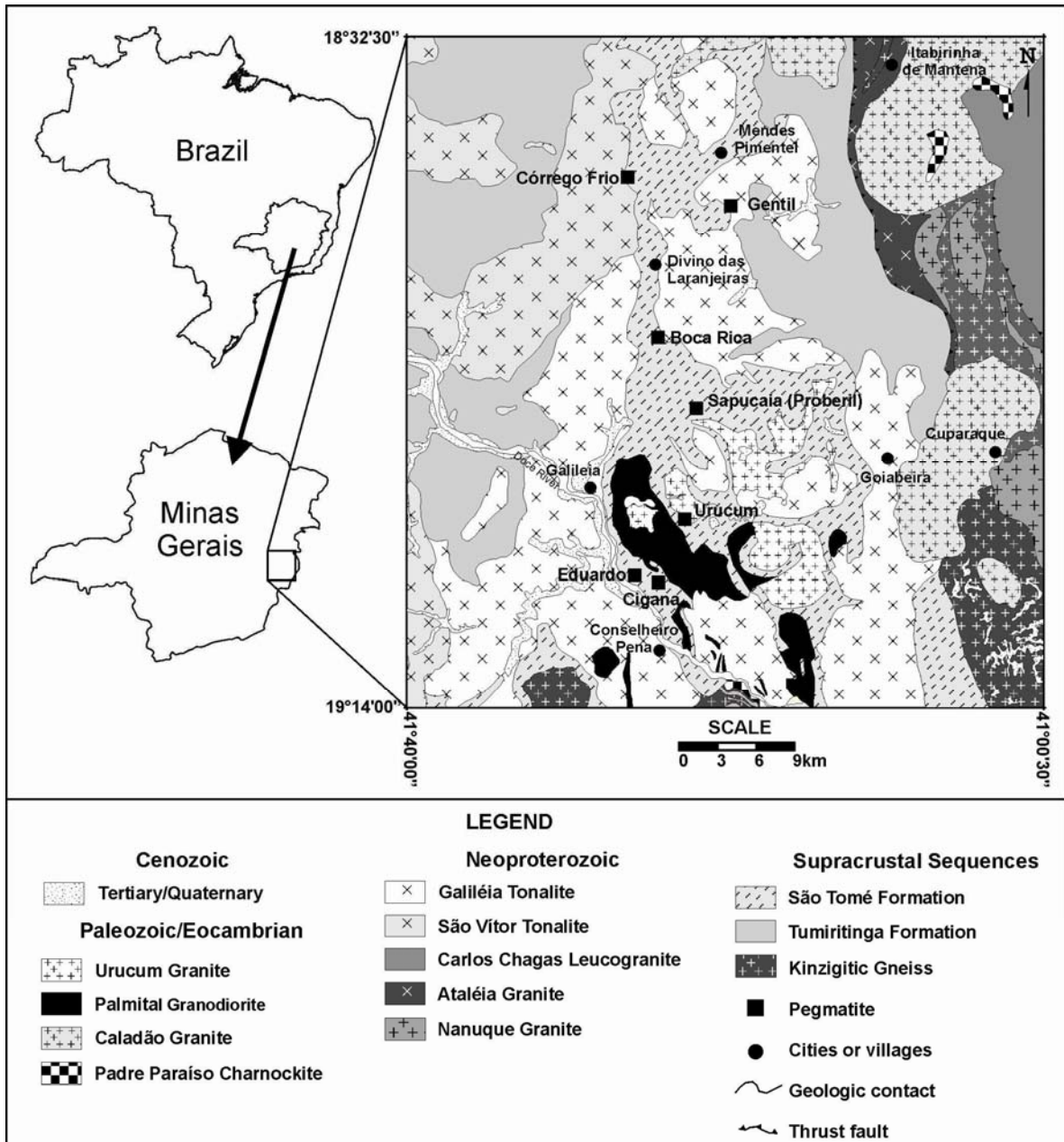
Table 4. Members of the laueite group

<i>M1</i>	<i>M2</i>	Phosphates	Arsenates
Mg	Fe <sup>3+</sup>	ushkovite	
Mn	Fe <sup>3+</sup>	laueite	
Fe <sup>2+</sup>	Fe <sup>3+</sup>	ferrolaueite	césarferreiraite
Fe <sup>3+</sup>	Fe <sup>3+</sup>		
Mg	Al	gordonite	maghrebite
Mn	Al	mangangordonite	
Fe <sup>2+</sup>	Al	paravauxite	
Fe <sup>3+</sup>	Al	sigloite	

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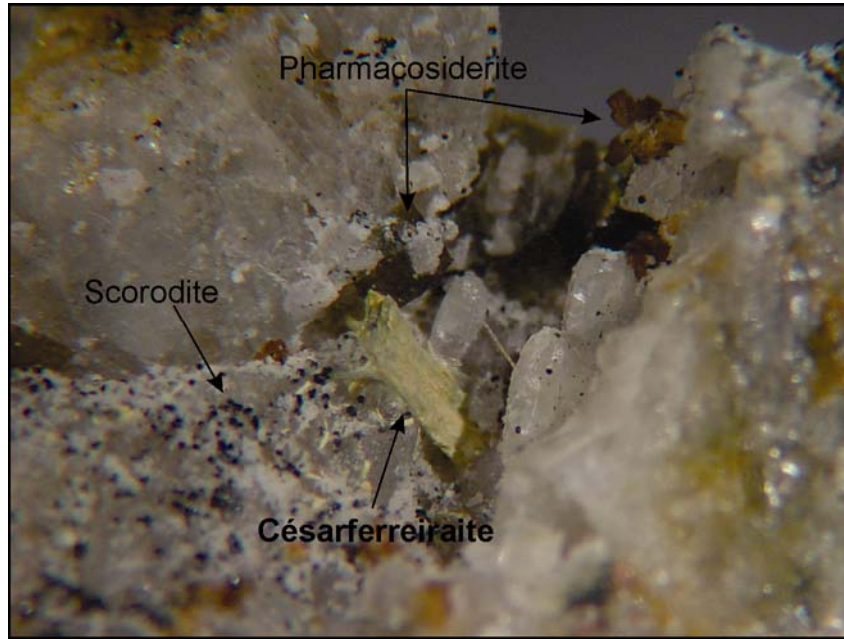
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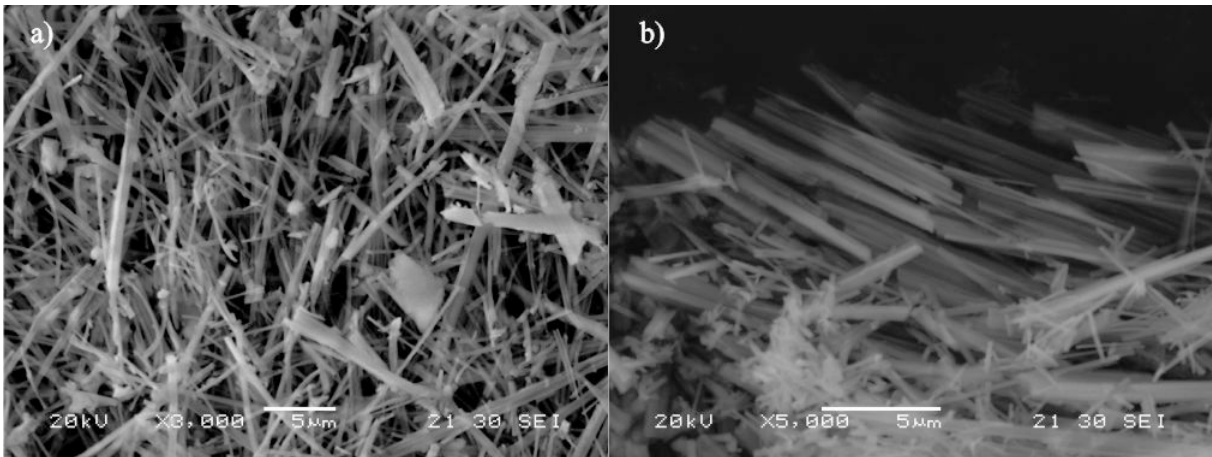
Figure 1 – Geological map of Conselheiro Pena Pegmatite Province and the location of the most important phosphate rich pegmatites (After Netto et al., 1998).





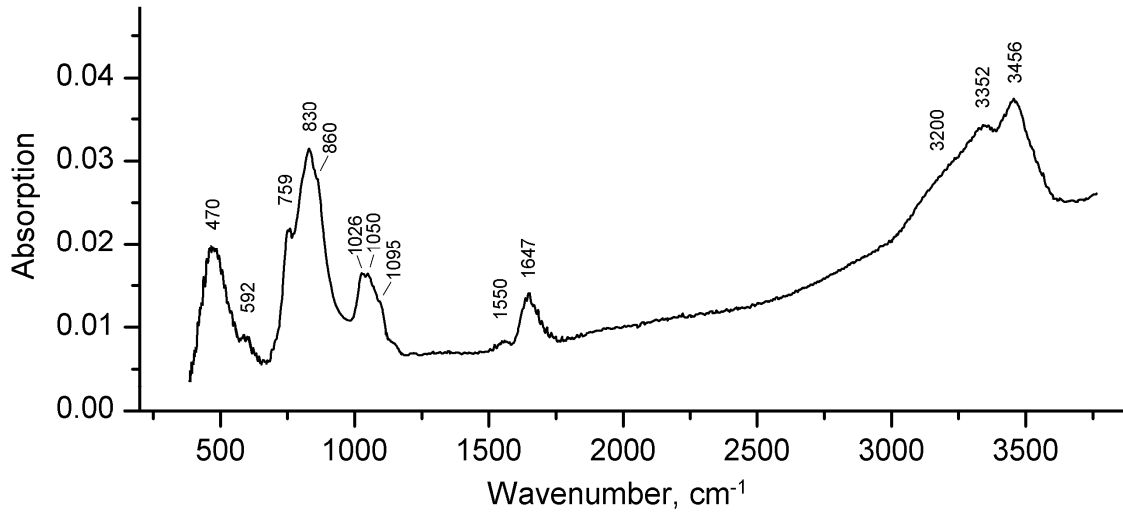
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Figure 2. 2 mm césarferreiraite crystal aggregate in association with pharmacosiderite and scorodite. Field of view: 1.0 cm.



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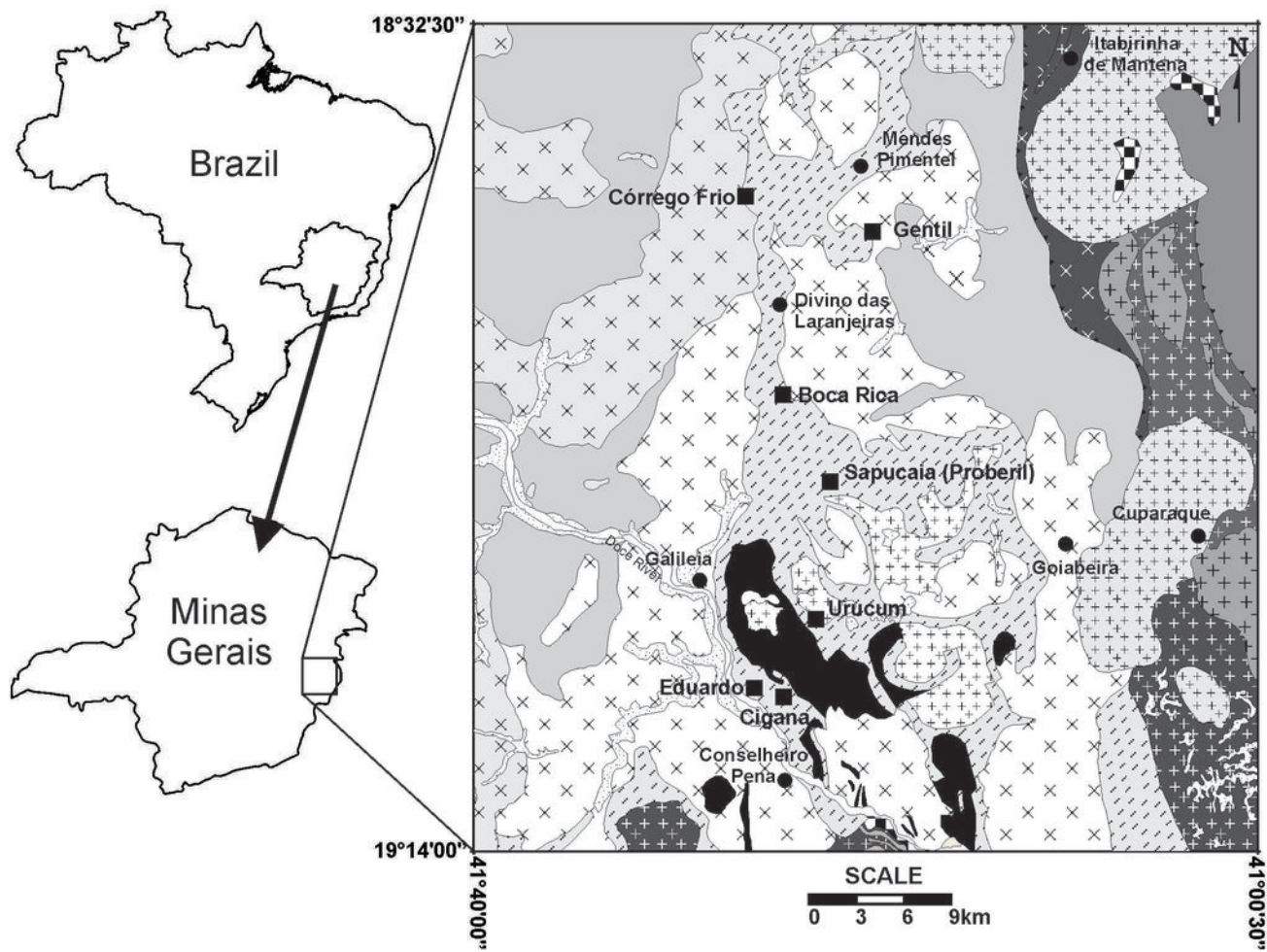
Figure 3. BSE-SEM images of césarferreiraite.



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Figure 4. The IR spectrum of Césarferreiraite.



### LEGEND

#### Cenozoico

Tertiary/Quaternary

#### Paleozoico/Eocambriano

Urucum Granite

Palmital Granodiorite

Caladão Granite

Padre Paraíso Charnockite

#### Neoproterozoico

Galiléia Tonalite

São Vítor Tonalite

Carlos Chagas Leucogranite

Ataléia Granite

Nanuque Granite

#### Supracrustal Sequences

São Tomé Formation

Tumiritinga Formation

Kinzigitic Gneiss

Pegmatite

Cities or villages

Geologic contact

Thrust fault

Pharmacosiderite

Scorodite

Césarferreiraite

