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# Geochronology of monazite related to REE, Nb-Ta and U-Th bearing minerals from Mesoproterozoic anorogenic magmatism in the E-Colombian Amazonian Craton: links to mantle plume activity in the Columbia (Nuna) supercontinent

José Alejandro Franco Victoria, Thomas Cramer, Alexandre de Oliveira Chaves, Heinrich Adolf Horn, Marc Poujol

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1 **Geochronology of monazite related to REE, Nb-Ta and U-**  
2 **Th bearing minerals from Mesoproterozoic anorogenic**  
3 **magmatism in the E-Colombian Amazonian Craton: links**  
4 **to mantle plume activity in the Columbia (Nuna)**  
5 **supercontinent**

6 Franco, José Alejandro<sup>1,2</sup>; Cramer, Thomas <sup>1,2</sup>, Chaves, Alexandre de Oliveira<sup>3</sup>; Horn,  
7 Heinrich Adolf<sup>2,3</sup>; Poujol, Marc<sup>2,4</sup>

- 8 1. *Universidad Nacional de Colombia, Bogotá, Colombia. Corresponding author e-mail:*  
9 *jafrancov@unal.edu.co*  
10 2. *Grupo de Estudios en Geología Económica y Mineralogía Aplicada (G.E.G.E.M.A)*  
11 3. *Universidade Federal de Minas Gerais (UFMG), Av. Antonio Carlos 6627, 31.270-901 Belo Horizonte*  
12 *– Brazil*  
13 4. *Univ Rennes, CNRS, Géosciences Rennes-UMR 6118, F-35000 Rennes, France.*  
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15

16 **Abstract**

17 U-Th-Pb geochronology of REE-bearing minerals such as monazite and other  
18 phosphates has become a precise tool to date mineralization events through  
19 geological time. In the E-Colombian Guainía department, from 3 colluvial sites  
20 over western Guiana Shield Mesoproterozoic rocks Nb-Ta, U-Th and REE bearing  
21 minerals probably of pegmatitic origin were sampled including up-to-0.5 cm long  
22 monazite crystals. The monazite pan concentrate samples from sites near to the  
23 indigenous communities of Barranquilla, San Jose and Chorro Bocón (Espina  
24 hills) along the Cuyari, Guainía, and Inírida rivers respectively, were analyzed  
25 geochemically and geochronologically using LA-ICP-MS and EPMA. The  
26 monazites obtained from the 3 sites are rather similar among each another and of  
27 approximate composition  
28  $Ce_{0.44}La_{0.16}Nd_{0.16}Sm_{0.04}Y_{0.03}Gd_{0.02}Dy_{0.01}Pr_{0.05}Th_{0.08}Pb_{0.01}Ca_{0.05}Si_{0.05}P_{0.91}O_{3.94}$ . Their  
29 EPMA and LA-ICP-MS ages suggest at least two mineralization events, a first one  
30 between 1528 and 1523 Ma in the Barranquilla and San Jose sites, related to  
31 rapakivi granites outcropping along the Cuyarí River and the Tabaquen rapakivi  
32 granite near the Guainía River, both emplaced ~1545 Ma ago. The second  
33 mineralization event from 1390-1343 Ma recorded in the Espina-hills is in the age  
34 range of the Matraca and Danta rapakivi granites that crystalized between 1400  
35 and 1340 Ma ago. These different anorogenic rapakivi granites were emplaced  
36 between 1550 and 1300 Ma into Mesoproterozoic basement rocks and were  
37 probably caused by intra-plate processes associated with mantle plume activity  
38 within the Columbia (Nuna) supercontinent.

39  
40 **Key words:** monazite; U-Pb and EPMA geochronology; anorogenic magmatism;  
41 mantle plume; Columbia (Nuna) supercontinent.

## 42 1 Introduction

43 Due to their high ionic charge and large ionic radii, Rare Earth Elements (REE)  
44 are not easily incorporated within the early-crystallizing silicates (Winter, 2001).  
45 Instead, they are enriched in highly differentiated magmatic phases together with  
46 other incompatible elements (such as P, K, Rb, U, Th). Despite similar chemical  
47 properties, they may be partitioned by either geochemical, mineralogical, and  
48 petrological processes in which case they become valuable proxies to study them  
49 (Henderson, 1984).

50 Generally spoken, Light Rare Earth Elements (LREE) tend to be concentrated in  
51 pegmatites and carbonatites, whereas Heavy Rare Earth Elements (HREE) are  
52 concentrated in granitic magmas (Pirajno, 2009).

53 Monazite is a LREE phosphate with the generalized formula  $(Ln_{III}, Th)(PO_4)$ ,  
54 where LnIII represents a trivalent light lanthanide, and is part of a complete solid  
55 solution with the LREE from La - Ce to Sm. Although present in most rock types,  
56 monazite is frequently found as an accessory mineral in low-Ca igneous rocks  
57 (Itano et al., 2016), instead of apatite and/or bastnäsite mineralization, and in  
58 differentiated magmatic phases like granites, syenites and granitic pegmatites. As  
59 monazite frequently occurs in colluvial and placer deposits, it is often better  
60 studied in these types of deposits than in the magmatic phases where they  
61 crystallized (Dill, 2010; Dill et al., 2012).

62 Elemental analyses of monazite can bring some information on its origin. For  
63 example, Itano et al. (2018) found that the La/Sm ratios in monazite grains from  
64 granite and granitic pegmatite and from magnetite and ilmenite magmas were  
65 different. This ratio could therefore be used as a good proxy for the type of magma.  
66 Although monazite can contain up to 12 wt% ThO<sub>2</sub> (Mottana et al., 1978) it is never  
67 metamict (Seydoux-Guillaume et al., 2018) and, such as zircon, usually does not  
68 incorporate initial lead when it crystallizes. Therefore, all the Pb comes from the  
69 radioactive decay of Th and U, which makes monazite a mineral suitable for U-  
70 Th-Pb dating via EPMA (electron probe micro analysis) (Itano et al., 2016) as  
71 described by Montel et al., (1996), Suzuki and Kato (2008) and Chaves et al.,  
72 (2013). This type of analyses should however be used with caution as, in some  
73 cases, EPMA dating can fail to resolve different monazite age populations as  
74 demonstrated by Poujol et al. (2017). Their use as geochronometers by means of  
75 LA-ICP-MS (Laser ablation inductively coupled plasma mass spectrometry) and  
76 EPMA is compiled by Engi (2017) and Williams et al. (2017).

77 Crustal-melt rapakivi granites with their characteristic coarse zoned feldspar  
78 megacrysts in a finer matrix are probably the result of mantle plume activity  
79 (Pirajno, 2007). In eastern Colombia, several authors have reported ages between  
80 1550 and 1340 Ma for anorogenic rapakivi granites such as the Parguaza, Danta,  
81 Matraca and Tabaquen intrusions (e.g. Bonilla et al., 2013, 2016; Cordani et al.,  
82 2016). In these granites, pegmatitic phases may host monazite, xenotime,  
83 columbite-tantalite, Nb-rich rutile as well as U oxides.

84 For this research, three localities in the E-Colombian Guainía Department were  
85 selected (Figures 1 and 2): Espina hills – near the Inírida river LOC (4), San José  
86 – near the Guainía river LOC (5) and Barranquilla – near the Cuyarí river LOC (6)  
87 where Mesoproterozoic pegmatite-related colluvial rock fragments contain  
88 centimetric Ti, Nb-Ta, U and Th bearing minerals together with monazite and  
89 xenotime. Here we present EPMA and LA-ICP-MS analyses performed on  
90 monazite crystals in order to determine their elemental compositions as well as  
91 their crystallization ages in order to understand their possible provenance within  
92 the western Guiana Shield, i.e. the NW-part of the Amazonian Craton.

93 Further we discuss pegmatitic phases with Ti, Nb-Ta, U-Th and REE  
94 mineralization in relation to the 1550-1340 Ma anorogenic granites found in  
95 various parts of the western Amazonian Craton in the light of intra-plate processes  
96 associated with mantle plume activity, which may have controlled this magmatism  
97 within the Columbia (Nuna) supercontinent.

## 98 **2 Methods used**

99 From panning concentrates (pc) more than 20 monazite crystals from each locality  
100 were picked and verified using a handheld Bruker Tracer III-V X-ray fluorescence  
101 instrument equipped with a Rh-tube and Silicon Drift Detector (SDD), working  
102 conditions 15-40kV, 5-10mA, 60-120s exposure time, no filter nor vacuum which  
103 together with the S1PXRf software allow to detect all elements heavier than Na  
104 (e.g. Magrini et al., 2018). Three epoxy resin pucks, labeled LOC (4), LOC (5) and  
105 LOC (6), each with ~10 crystals, were prepared using buehler epoxy cure2 resin  
106 and hardener, polished first with silicon carbide and diamond and then coated with  
107 graphite (Mange and Wright, 2007; Reed, 2005). Scanning electron microscopy  
108 (SEM) BSE images of the monazite crystals allowed choosing the most  
109 homogeneous zones, free of inclusions or in very low proportion. Their chemical  
110 composition was determined using wavelength-dispersive spectroscopy (WDS) in  
111 a JEOL JXA-8900 EPMA in the Laboratory of Microanalysis of the Microscopy  
112 Center at Federal University of Minas Gerais (LMA-CM-UFMG), Brazil. The  
113 reference materials used were Y<sub>2</sub>O<sub>3</sub> (YAG\_25K50n), Dy<sub>2</sub>O<sub>3</sub> (DyPO<sub>4</sub>\_25K50n),  
114 P<sub>2</sub>O<sub>5</sub> (Monaz\_25K50n), SiO<sub>2</sub> (ThSiO<sub>4</sub>\_25K50n), Gd<sub>2</sub>O<sub>3</sub> (GdPO<sub>4</sub>\_25K50n), PbO  
115 (Crocoi\_25K50n), Sm<sub>2</sub>O<sub>3</sub> (REE<sub>2</sub>\_25K50n), ThO<sub>2</sub> (ThSiO<sub>4</sub>\_25K50n), Nd<sub>2</sub>O<sub>3</sub>  
116 (NdPO<sub>4</sub>\_25K50n), UO<sub>2</sub> (UO<sub>2</sub>\_25K50n), Pr<sub>2</sub>O<sub>3</sub> (PrPO<sub>4</sub>\_25K50n), CaO  
117 (Ca<sub>2</sub>P<sub>2</sub>O<sub>7</sub>\_25K50n), (Ce<sub>2</sub>O<sub>3</sub>) (Monaz\_25K50n) and La<sub>2</sub>O<sub>3</sub> (Monaz\_25K50n).  
118 Additionally, 30 monazite crystals up-to-0,5 cm large from LOC (5) were analyzed.  
119 For EPMA dating, from each locality 1 monazite crystal from the polished epoxy  
120 pucks was selected, and ages were calculated using U, Th and Pb concentrations  
121 (Williams et al., 2007).

122 The EPMA-analyses were performed in 6 to 10 spots per crystal. Standards and  
123 quantitative WDS analytical parameters used during monazite analyses are  
124 described in Chaves et al. (2013). Overlaps of X-ray peaks between Y and Pb  
125 have not been corrected because there was no measurement in PbMa (Lead M

126 alpha), but only in PbMβ (Lead M beta). However, to avoid errors in the obtained  
127 ages, the interference of ThMγ (Thorium M gamma) on the measured U<sub>Mβ</sub>  
128 (Uranium M beta) had to be corrected, following Scherrer et al. (2000) in  
129 adaptation to the conditions of the LMA-CM-UFMG, as follows (Chaves et al.  
130 2013):

131  $U_{\text{corrected}} = U_{\text{measured}} - (0.006365 \times Th_{\text{measured}})$ .

132 Microprobe chemical U-Th-Pb<sub>T</sub> ages and associated errors were obtained using  
133 EPMA Dating software (Pommier et al., 2004) and the average ages were taken  
134 through Isoplot/Ex software (Ludwig, 2003). The age equation used is:

$$135 \quad Pb = \{Th \times [\exp(\lambda_{232}T) - 1] \times (M_{208} / M_{232})\} + \{U_c \times [\exp(\lambda_{238}T) \\ 136 \quad \quad \quad - 1] \times (M_{206} / M_{238}) \times 0.9928\} + \{U_c \times [\exp(\lambda_{235}T) - 1] \\ 137 \quad \quad \quad - 1] \times (M_{207} / M_{235}) \times 0.0072\}$$

138

139 Where:  $^{238}U / (^{235}U + ^{238}U) = 0.9928$  and  $^{235}U / (^{235}U + ^{238}U) = 0.0072$ .  $U_c$  (U corrected),  
140 Th and Pb are concentrations in ppm; T is the age in Ma;  $M_{206}$ ,  $M_{207}$ ,  $M_{208}$ ,  
141  $M_{235}$ ,  $M_{238}$ ,  $M_{232}$  are the atomic masses of  $^{206}Pb$ ,  $^{207}Pb$ ,  $^{208}Pb$ ,  $^{235}U$ ,  $^{238}U$ ,  $^{232}Th$ ;  
142  $\lambda_{232} = 0.49475 \times 10^{-4} \text{ Ma}^{-1}$ ;  $\lambda_{238} = 1.55125 \times 10^{-4} \text{ Ma}^{-1}$ ;  $\lambda_{235} = 9.8485 \times 10^{-4} \text{ Ma}^{-1}$   
143 (Pommier et al., 2004).

144 Ages and uncertainties were obtained using EPMA Dating software (Pommier et  
145 al., 2004) and age averages were processed using Isoplot 3.75 Excel version  
146 (Ludwig, 2012, 2003).

147 Beside the U and Pb values, also the other elements were used for unravelling  
148 both mineral chemistry and REE-geochemistry and calculate approximate mineral  
149 compositions assuming an idealized monazite formula  $Ln^{3+}PO_4$  and considering  
150 all elements as part of the mineral lattice (i.e. not specifying possible inclusions or  
151 exsolutions of other phases). For the formulae calculations, mineralogical  
152 standard normalization procedures were applied (Deer et al., 2013; Perkins,  
153 2014), i.e. the transformation of the mean oxide wt-% to mole-%, the distribution  
154 of the cations to the four anionic O-sites, and the calculation if the cation and anion  
155 charges became balanced.

156 LA-ICP-MS dating has been performed by using aleatory points in all crystals of  
157 the three sampling sites LOC (4, 5 and 6) including the same crystals analyzed by  
158 EPMA. Analyses were carried out on a single quadrupole Agilent 7700 ICP-MS  
159 mass spectrometer coupled to a 193-nm excimer laser system ESI (NWR193UC)  
160 in the GeOHeLiS analytical platform of the University of Rennes 1 in France. In  
161 the present study, the acquisition time of each analytical spot was set to 80  
162 seconds (20 seconds background followed by 60 seconds with the laser firing).  
163 Spots diameters of 8  $\mu\text{m}$  associated with repetition rates of 3 Hz with a laser  
164 fluency of  $5 \text{ J.cm}^{-2}$  were used. The signal of  $^{204}(Pb+Hg)$ ,  $^{206}Pb$ ,  $^{207}Pb$ ,  $^{208}Pb$ ,  $^{232}Th$   
165 and  $^{238}U$  masses were acquired. The  $^{235}U$  signal is calculated from  $^{238}U$  based on  
166 the ratio  $^{238}U / ^{235}U = 137.818$  (Hiess et al., 2012).

167 Data were corrected for U–Pb and Th–Pb fractionation and for the mass bias by  
168 standard bracketing with repeated measurements of the Moacir monazite  
169 reference material (Gasquet et al., 2010). Data reduction was carried out with the  
170 software package GLITTER® (Van Achterbergh et al., 2001). Repeated analyzes  
171 of the Manangoutry monazite reference material (Paquette and Tiepolo, 2007)  
172 treated as unknown were used to control the reproducibility and accuracy of the  
173 corrections and yield a concordia age of  $548 \pm 6$  Ma (MSWD=0.98; n=11). After  
174 data reduction, U-Th-Pb Concordia diagrams were constructed with the IsoplotR  
175 software (Vermeesch, 2018). The calculated Concordia ages are quoted with 95%  
176 of confidence interval. More information on the analytical protocol in Poujol M, et  
177 al., (2017) and supplementary table in Annex. A.

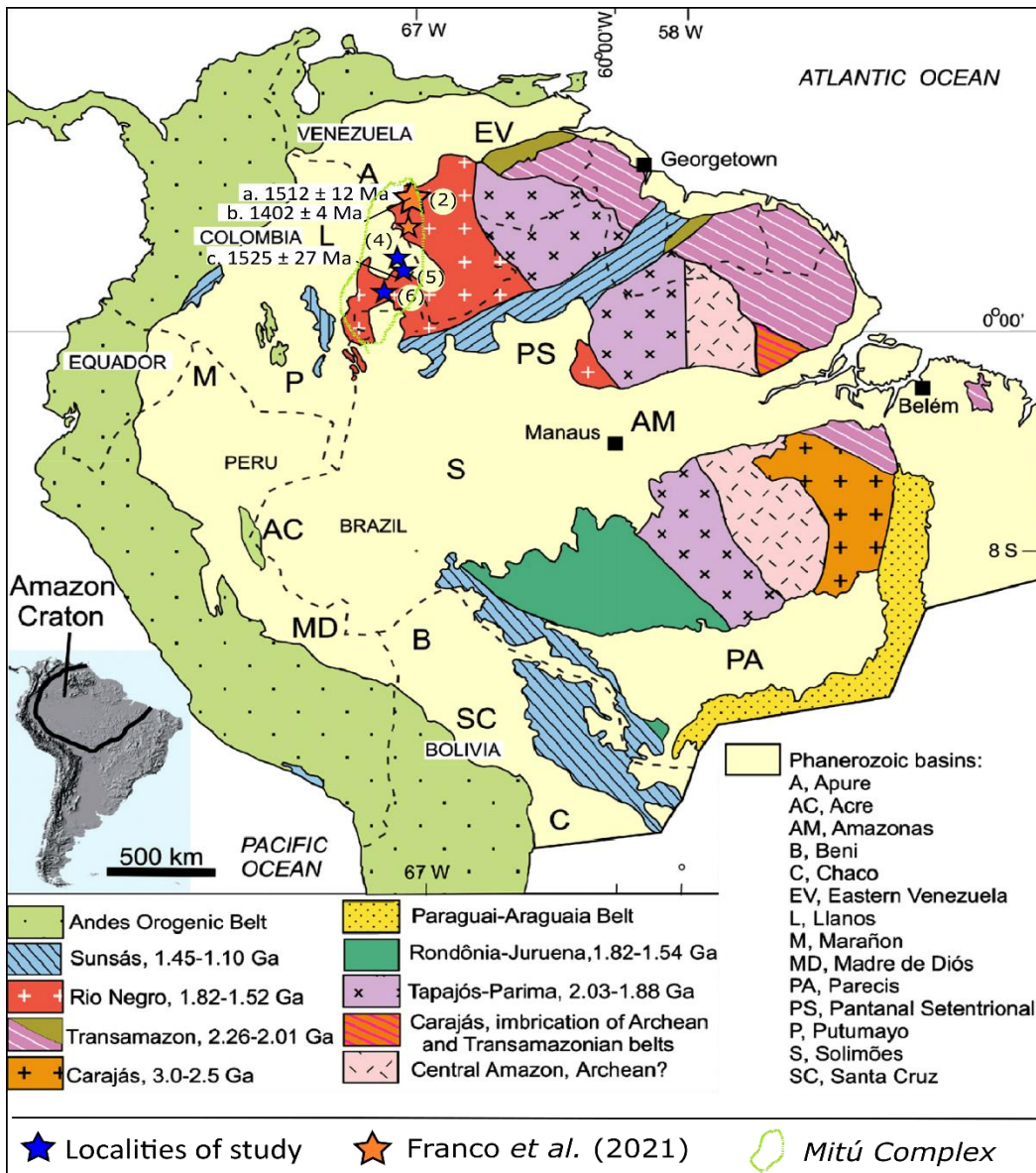
178

### 179 **3 Geological background**

180 The Guainía Department is located at the northwestern part of the Guiana Shield  
181 i.e. a portion of the Colombian part of the Amazon Craton, which extends from the  
182 Andean Eastern Cordillera foothills to Venezuela and Brazil and is the least  
183 populated and studied area of Colombia. Our geological knowledge of this area is  
184 largely based on research conducted in Brazil and Venezuela (e.g., Gansser,  
185 1954, 1974; Amaral, 1974; Gaudette et al., 1978; Almeida et al., 1981; Gaudette  
186 and Olszewski, 1985; Bettencourt et al., 1999; Tassinari et al., 1999 and (Santos  
187 et al., 2000), although important studies have been done recently also in Colombia  
188 (e.g., Bonilla et al., 2013, 2016, 2019; Cordani et al., 2016, Ibanez et al., 2011,  
189 Kroonenberg, 2019).

190 According to Santos (2003), the crystalline basement in the Western Guiana  
191 Shield is linked to the Rio Negro geochronological province 1.82 – 1.52 Ga (Figure  
192 1).

193



194

195 Figure 1. Regional map showing the geochronological provinces and some ages in Ma  
 196 reported for rocks and minerals in and outside the study area: a. U-Pb in rutile (Nb,Ta)  
 197 from Cachicamo hills; b. U-Pb in zircon from the basement zone (Bonilla et al., 2013); c.  
 198 U/Pb in zircon from granitic rocks of Tabaquen in the Guainía River Bonilla (2019).  
 199 Modified from Santos et al. (2008).

200 Enhanced by the PRORADAM program (Galvis Vergara et al., 1979a; Huguett et  
 201 al., 1979) the complex geological history of Colombia since the Proterozoic has  
 202 been slowly revealed, with different phases of extension and compression,  
 203 orogenesis, erosion, sedimentation, metamorphism and emplacement of a wide  
 204 variety of magmatic rocks. Hallmarks in descriptions represent Celada et al.  
 205 (2006) and recently Kroonenberg (2019) synthesized the actual geological  
 206 information of E-Colombia.

207 Different orogenic episodes in the Guiana Shield have been recognized so far  
208 (Kroonenberg 2019): the Transamazonian Orogeny (2.26 to 1.98 Ga) in the  
209 Eastern margin (as result of the collision with Proto-Africa); the Querarí Orogeny  
210 (1.86–1.72 Ga) at the Western border between Colombia, W-Venezuela and NW-  
211 Brazil; and, in the framework of the Rodinia supercontinent formation, the  
212 Grenvillian Orogeny (1.3–1.0 Ga), called Putumayo Orogeny by Ibáñez-Mejía et  
213 al. (2011) as revealed by some inliers in the Andes but also eastward by gabbroic  
214 intrusions (Bonilla Perez et al., 2020).

215 In the Guainía Department between the Guaviare River to the north, the Caquetá  
216 River to the south, and the Chiribiquete mountain range to the west only very few  
217 outcrops have been studied in the past. The oldest rocks seem to be felsic to  
218 intermediate metavolcanic rocks along the Atabapo River and parts of the Río  
219 Negro River, which Galvis et al. (1979) and Huguett et al. (1979) named “Neises  
220 del Atabapo-Río Negro” (see López et al., 2010). Kroonenberg (2019) proposed  
221 that they do not belong to the Mitú-Complex but rather to the Mid-  
222 Paleoproterozoic Caicara Metavolcanics and associated them with the  $1984 \pm 9$   
223 Ma Surumu Rhyodacite (SHRIMP U-Pb in zircons, Santos et al., 2003).

224 The Colombian basement was first described as the “Basement Group” (Gansser,  
225 1954) or as the “Guyanese Complex” (Pinheiro et al., 1976), then as the Migmatitic  
226 Mitú Complex (Galvis Vergara et al., 1979b) and later as the Mitú Complex (López  
227 Isaza, 2012; López Isaza et al., 2007; López Isaza and Cramer, 2012). Its  
228 extension has been initially inferred based on geophysical data. Most of this  
229 basement is constituted by Precambrian crystalline rocks. It is covered by  
230 Cenozoic sediments, as well as by a thick tropical forest covering peneplanes  
231 modeled by strong tropical weathering and meandering rivers in extensive alluvial  
232 plains, locally interrupted by inselbergs of the old basement.

233 The Mitú Complex is composed of high-grade metamorphic rocks and granitoids  
234 with various compositions and affinities, formed during different orogenic events  
235 (Kroonenberg, 2019; López Isaza et al., 2007). The metamorphosed igneous and  
236 sedimentary protoliths appear mainly as quartz feldspathic gneisses with alaskite  
237 to monzonite composition, but also as amphibolite, quartzite, metagranitoids,  
238 calco-silicate (rich in epidote group minerals) and quartz gneisses, some of them  
239 with migmatitic structures.

240 Large parts of the Mitú Complex (Figure 2) are composed of monzogranites with  
241 calc-alkaline affinities, besides meta- to peraluminous granites (U-Pb in zircon LA-  
242 MC-ICP-MS Concordia age of  $1574 \pm 10$  Ma for the Mitú granite; Ibáñez-Mejía et  
243 al., 2011), as well as of other granites. Among the clearly metamorphic rocks,  
244 quartz-feldspathic gneisses are the most abundant with U-Pb zircon ages  
245 between 1800-1760 Ma (Bonilla et al., 2019; Cordani et al., 2016; Kroonenberg,  
246 2019; López et al., 2007). Medium grade amphibolite facies metamorphic rocks  
247 within a series of essentially juvenile magmatic arcs in the Rio Negro Juruena  
248 province are interpreted by several workers as subduction-related (Cordani et al.,  
249 2016; Tassinari and Macambira, 1999).



250 Younger granitoids in the southeastern part (especially Guainía Department)  
251 exhibit variable compositions, porphyritic textures and may contain feldspar  
252 phenocrysts. They were emplaced ca. 1750 Ma ago during the Statherian (Bonilla  
253 et al., 2019) while some two-mica granite intrusions are 1600-1550 Ma old (Bonilla  
254 et al., 2019). It must be noticed that other ~1600 – 1500 Ma old granites have  
255 been described along the whole Colombian eastern territory (Cordani et al., 2016;  
256 Ibañez, 2010; Priem et al., 1982; Rodriguez et al., 2011).

257 The Parguaza batholith, emplaced 1550-1500 Ma ago, represents a primary  
258 source of Sn, Nb, Ta and REE mineralization (Aarden and Davidson, 1977;  
259 Herrera-Bangerter, 1989; Mariño, 2012; 2013) although it is largely covered by  
260 partially lateritic Cenozoic sediments in plains and valleys. In the Venezuelan  
261 localities of El Burro, La Fortuna and Aguamena, colluvial - alluvial deposits  
262 contain sub-to economic concentrations of minerals such as cassiterite, rutile-  
263 (Nb,Ta), columbite-tantalite and REE minerals.

264 An important feeder of rare element minerals in the Parguaza Batholith seem to  
265 be pegmatites, and similarly pegmatites associated to the Mitú Complex (e.g.  
266 López Isaza and Cramer, 2012) may also be the main primary source of the  
267 monazites and xenotimes discussed here.

#### 268 **4 Pegmatites**

269 Due to their economic importance and variability, manifold pegmatite classification  
270 efforts (e.g. Ginsburg et al., 1979) were developed over time which are still  
271 controversial and object of research. Thus Černý (1991) proposed the existence  
272 of two main pegmatite families, the LCT (Li-Cs-Ta) and the NYF (Nb-Y-F), a  
273 concept however only applicable for highly fractionated pegmatites if exotic  
274 minerals such as spodumene, tantalite, pyrochlore, pollucite, etc. are present  
275 (London, 2018). Černý and Ercit (2005) and Černý et al. (2012) defined, based on  
276 metamorphic environment, mineralogy, elemental composition and texture, five  
277 pegmatite classes from which four may host Ta–Nb–Sn mineralization (especially  
278 the abyssal, muscovite-rare-element, rare-element and miarolitic classes) and 10  
279 subclasses. The rare-element class is the most important for Nb–Ta  
280 mineralization, which are further subdivided depending on major and minor  
281 minerals carrying Li, Be, REE and Nb–Ta.

282 Most of the pegmatites with the LCT signature have compositional affinity with S-  
283 type granites (Chappell and White, 1974, 2001). Their protoliths are assumed to  
284 be pelitic marine sediments as source of Al - and trace elements enrichment like  
285 B, Be, P and Sn. From those after anatexis melting of metamorphic schists and  
286 gneisses, reduced granites and derived specialized pegmatites could form with  
287 high contents in Li, Cs, Ta where fluxing components like B, P, and F play an  
288 important role. Many of these trace elements with different grades of  
289 incompatibility are contained in micas and feldspars and liberated during the melt-  
290 forming reactions and may concentrate furthermore in specialized pegmatites  
291 above the mineralization limit, i.e. forming abundant and large visible minerals

292 characteristic of many famous and economic important pegmatite ore deposits  
293 worldwide.

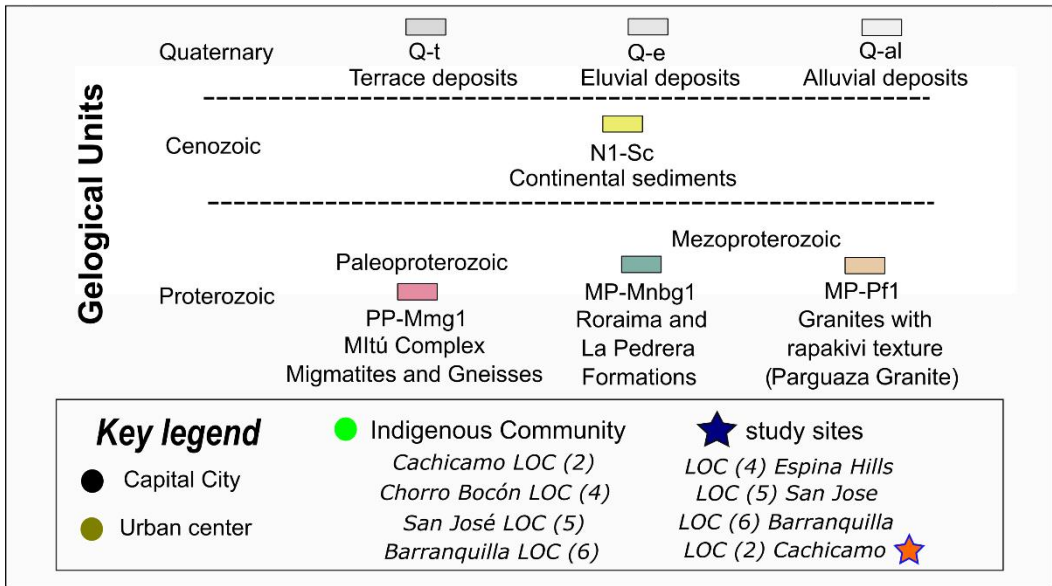
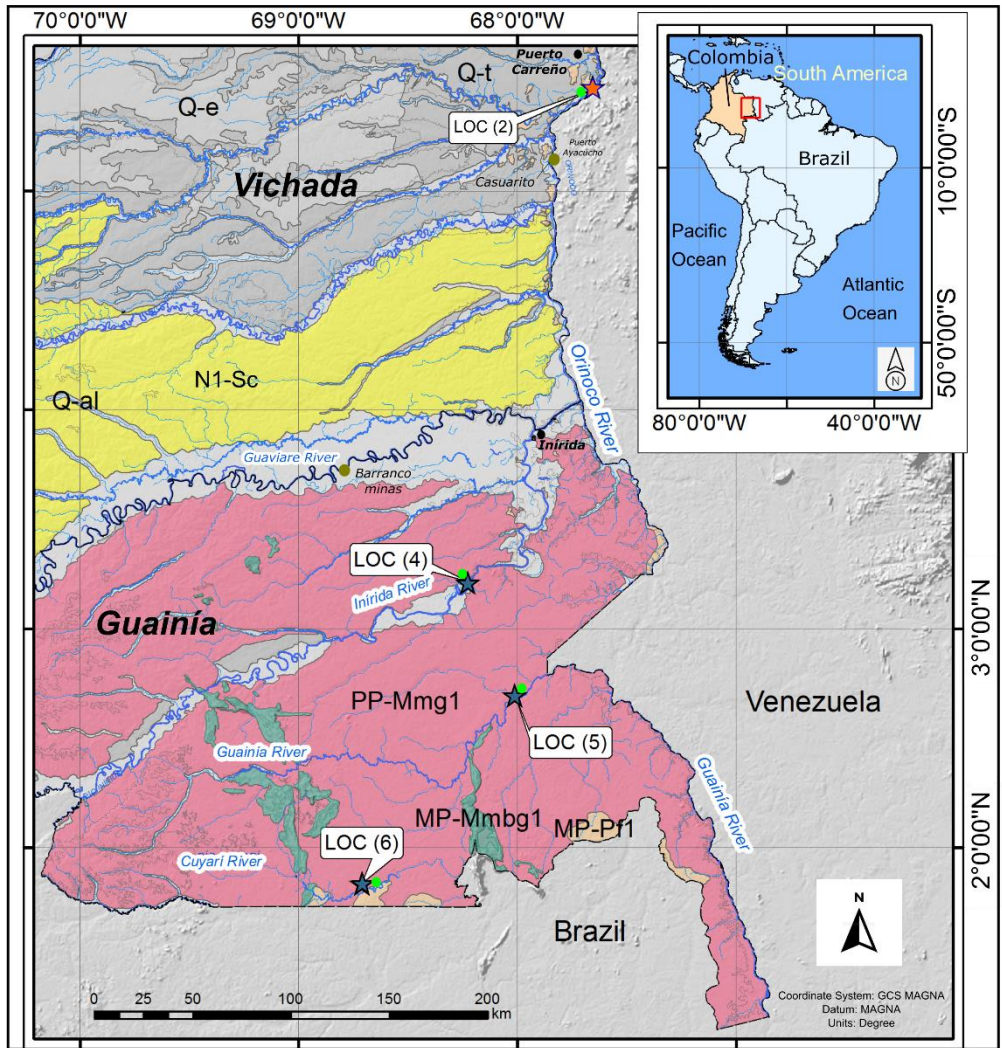
294 On the other hand, most of the NYF-family pegmatites are thought to be derived  
295 from A-type granites, where “A” may mean “anorogenic” (e.g. Eby, 1990) but also  
296 Al-rich or abnormal and their origins are debatable. For example, Frost and Frost  
297 (2014) emphasized their higher Fe-content and proposed to name them ferroan  
298 alkali-calcic to calc-alkalic granitic rocks. Their source may be gneissic granulites  
299 deep in the continental crust, with some mafic mantle contribution or low-density  
300 carbonic fluid. The NYF-family contains chemically complex oxides and silicates  
301 that carry heavy rare earth elements (HREE), Ti, U, Th, and Nb > Ta (Cerný et al.,  
302 2012). Abundant fluorite or topaz reflects the enrichment in fluorine. The NYF  
303 pegmatites are depleted in phosphorus, and tourmaline is uncommon.

304 Subduction-related I-type granites may be a smaller source both of LCT and NYF  
305 pegmatites (Černý and Ercit, 2005), but there are a lot of pegmatites worldwide,  
306 which may or may not fit clearly in any of these models (Bonin, 2007; Cerný et al.,  
307 2012; Dall’Agnol et al., 1999; Dill, 2015; Dill et al., 2006; Gandhi and Sarkar, 2016;  
308 Kroonenberg et al., 2016; Laznicka, 2006; London and Morgan, 2012; Sial et al.,  
309 2011).

310

## 311 **5 Selected mineral occurrences**

312 The Guainía region is mostly covered by humid tropical forests and extensive  
313 alluvial plains with preferably west-east drainage of the main rivers Guaviare,  
314 Inírida, Guainía and Cuyarí to the Orinoco and Amazonas rivers, which constitute  
315 also the main ways of communication between local indigenous communities and  
316 non-local population. During several fieldtrips, sediments and basement rocks  
317 outcropping along the main rivers including inselbergs like Mavicure, Espina,  
318 Tortuga, Tabaquen, Matraca and Danta were sampled, and from Vichada to  
319 Guainía departments, four localities from north to south were defined for more  
320 detailed studies and are described elsewhere, especially the Cachicamo rutilite-  
321 (Nb), closely associated to the Parguaza Batholith (Franco et al., 2021). For this  
322 study, we analyzed three of them in the Guainía Department (Figure 2): LOC (4)  
323 near to the Inírida River and the Espina hills, LOC (5) near to San Jose at the  
324 Guainía River, and LOC (6) near to Barranquilla at the Cuyari River.



325

326  
327

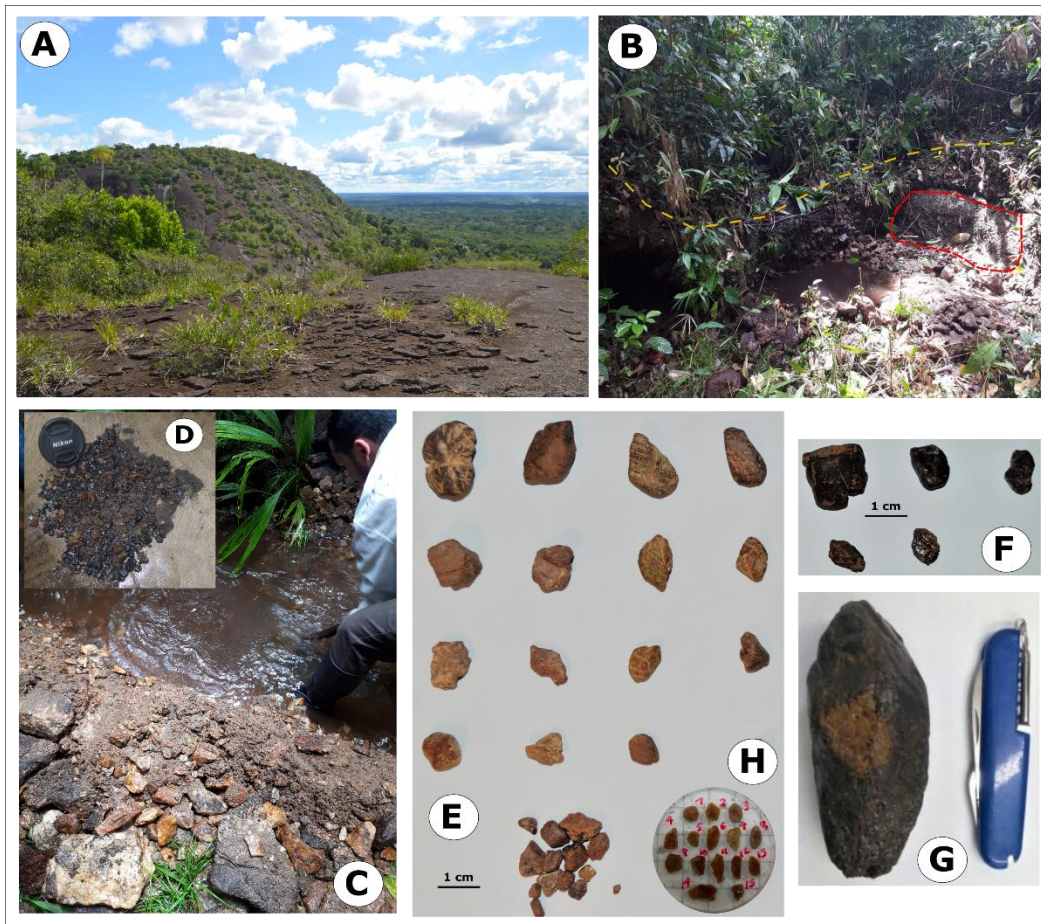
Figure 2. Generalized geological map of Vichada and Guainía Department, eastern part of Colombia showing the localities (4, 5 and 6) (blue stars) and locality (2) (pink star) -

328 from which localities (4 to 6) were selected for this study; locality (2) associated to the  
329 Parguaza Granite was studied by Franco et al., (2021) and is here shown for a better  
330 overview. As green dots the names of the communities nearby the sampling locations.  
331 Modified from the Geological Map of Colombia (Gómez Tapias et al., 2015).

332

### 333 5.1 Monazite, xenotime and Nb, Ta, U, Zr-minerals from Espina hills - 334 Inírida River LOC (4)

335 In the Espina foothills as main mineralization site of LOC (4) N 03° 12' 13.6", W  
336 068° 09' 19.6" (Figure 2), monazite occurs in colluvial deposits with large blocks  
337 of quartz and muscovite pegmatites. Monazite crystals can reach up to 1 cm in  
338 length and are associated with rutile-(Nb,Ta), xenotime, REE-enriched zircon and  
339 U-Th bearing minerals and, in some cases, black tourmalines in the panning  
340 concentrates (Figure 3).



341

342 Figure 3. Pictures of field and mineral found in panning concentrates from LOC (4) Espina  
343 Hills. A. view from the top of Espina Hills; B. colluvial deposits in the foothill of Espina Hills  
344 (yellow line shows the limit of the dissected colluvium and red line basement rock); C and  
345 D. sample site and heavy mineral concentrate and pan concentrate, respectively; E.  
346 Monazite and xenotime crystals selected from the pan concentrates; F. U-Th bearing  
347 minerals; G. Rutile-(Nb) associated to monazite (brown color) and H. Polished epoxy resin  
348 puck of monazite for analysis.

349 **5.2 Monazite and Ta, Nb-minerals at the San Jose - Guainía River**  
350 **LOC (5)**

351 Near to the Indigenous community of San José labelled LOC (5) N 02° 41' 59.6",  
352 W 068° 01' 38.4" (Figure 2), monazite grains were found in the regoliths overlying  
353 pegmatitic dykes emplaced in gneiss and migmatites of the Mitú Complex.  
354 However, the most representative crystals occur in colluvium deposits together  
355 within blocks and gravels of quartz and muscovite dyke fragments along the Seje  
356 and Lombriz creeks. Monazite crystal can reach up to 3 cm in length and are found  
357 together with Fe-columbite, rutile-(Nb,Ta) and magnetite (Figure 4). Occasionally  
358 isolated xenotime and tourmaline crystals are found in the panning concentrates.

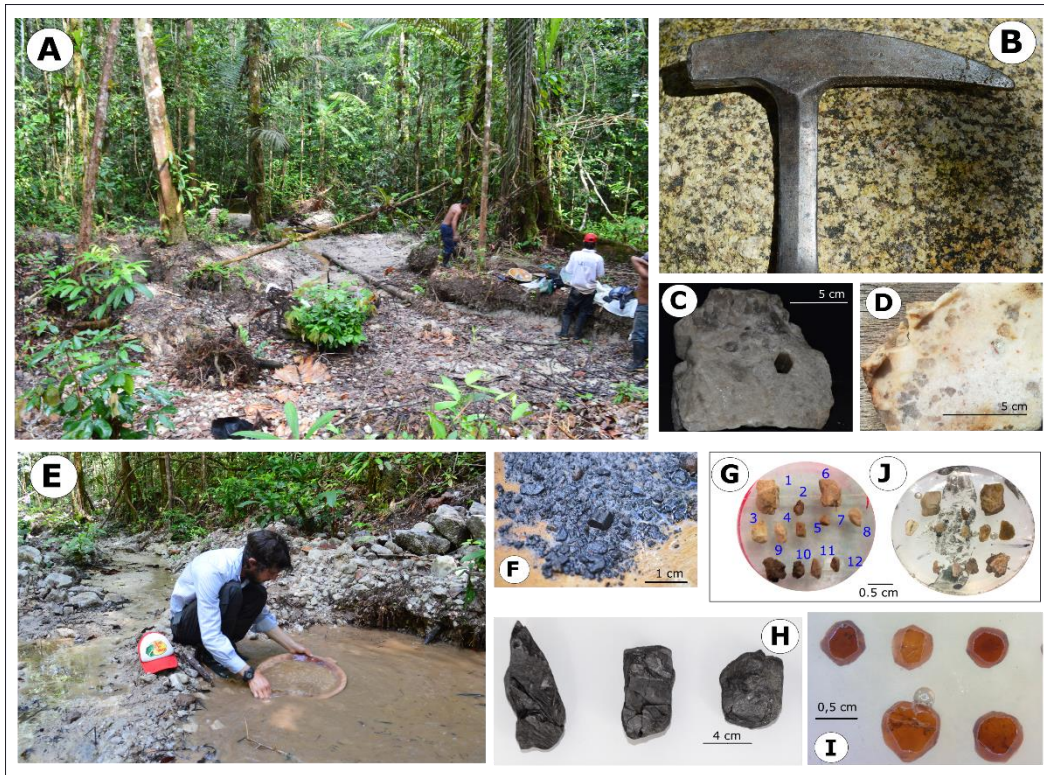


359  
360 Figure 4. Pictures of field and mineral found in panning concentrates from LOC (5) San  
361 Jose. A. view of rocks of Mitú complex in the Guainía River at San Jose indigenous  
362 community; B. Pegmatite with an elevated radioactivity measures; C. Pegmatite with Fe-  
363 columbite crystal; D. Sample site of heavy mineral concentration E. Fe-columbite crystals  
364 from pan concentrates; F; Huge monazite crystals from pan concentrates; G. Monazite  
365 crystals selected from the pan concentrates and H. Polished epoxy resin puck of monazite  
366 for analysis.

367 **5.3 Monazite and Ta, Nb, Zr and REE-minerals from Barranquilla -**  
368 **Cuyari River LOC (6)**

369 Monazite grains were found near Barranquilla labelled LOC (6) N 01° 48' 51.9",  
370 W 068° 41' 12.5" (Figure 2), in colluvium deposits cut by small drains along the

371 Cuyari River basin, together with smoky quartz blocks, muscovite, beryl and Fe-  
 372 columbite dikes as well as K-feldspar, quartz and garnet bearing dikes. They are  
 373 related to plutonic rocks from syeno-granites to acidic granites, some of them with  
 374 rapakivi texture. Monazite can reach up to 0,5 cm in length and are found together  
 375 with Fe-columbite, Mn-Tantalite, rutile-(Nb,Ta), xenotime, zircon, and garnet  
 376 (spessartine) (Figure 5). Isolated crystals of black tourmaline occasionally occur  
 377 in panning concentrates.



378  
 379 Figure 5. Pictures of field and mineral found in panning concentrates from LOC (6)  
 380 Barranquilla. A. Colluvial deposits explored by local people from Barranquilla indigenous  
 381 community; B. Block of two mica granite founded in the base of colluviums; C. Pegmatite  
 382 with a retaining the shape of a beryl crystal y D Sample of K-feldspar - garnet pegmatite;  
 383 E. Sample site of heavy mineral; F. Heavy mineral concentrate; G. Monazite crystals  
 384 selected from the pan concentrates (1-5); H. Columbite-tantalite crystals; I. Garnet crystals  
 385 and J. Polished epoxy resin puck of monazite for analysis.

## 386 6 Results

### 387 6.1 Monazite chemistry

388 The pXFR-measurements were in good qualitative agreement with the EPMA data  
 389 (supplementary Table in Annex A) which reveal that all the monazite crystals are  
 390 Ce and La dominated. As the BSE-images Figure 7 show very inclusion-poor  
 391 monazites, the calculated mineral formulae should be very near to the real  
 392 monazite compositions. The HREE Ho, Er, Tm, Yb and Lu were below EPMA-  
 393 detection limit. In LOC (4), monazite grains are very homogeneous with  $Ce_2O_3$   
 394 between 29 and 31% and  $ThO_2$  between 8 and 9 %. The standard deviation

395 among the 7 measured monazite crystals of the main cations is below 0,1%. Their  
396 average formula including all elements could be expressed as  
397  $Ce_{0.44}La_{0.16}Nd_{0.16}Sm_{0.04}Y_{0.03}Gd_{0.02}Dy_{0.01}Pr_{0.05}Th_{0.08}Pb_{0.01}Ca_{0.05}Si_{0.05}P_{0.91}O_{3.94}$ .

398 Monazite crystals from LOC (5) show a more heterogeneous composition with  
399  $Ce_2O_3$  between 27 and 34% and  $ThO_2$  between 4 and 13 % resulting in standard  
400 deviations (n=8) of 7% for Th, and around 1% for La, Ce, Sm and Ca.

401 Their average formula would be  $Ce_{0.44}La_{0.16}Nd_{0.14}Pr_{0.04}Sm_{0.04}Y_{0.03}Gd_{0.02}$   
402  $Dy_{0.01}Th_{0.09}Ca_{0.07}Pb_{0.01}Si_{0.05}P_{0.91}O_{3.94}$ .

403

404 In LOC (6), monazite grains are still less heterogeneous than in LOC (4) and LOC  
405 5 with  $Ce_2O_3$  between 26 and 29% and  $ThO_2$  between 9 and 12 %. The REE  
406 standard deviations (n=10) are around or below 0,1%. Their average formula  
407 would be:

408  $Ce_{0.42}La_{0.13}Nd_{0.19}Pr_{0.05}Sm_{0.05}Y_{0.05}Gd_{0.02}Dy_{0.01}Th_{0.10}Ca_{0.04}Pb_{0.01}Si_{0.08}P_{0.86}O_{3.94}$ .

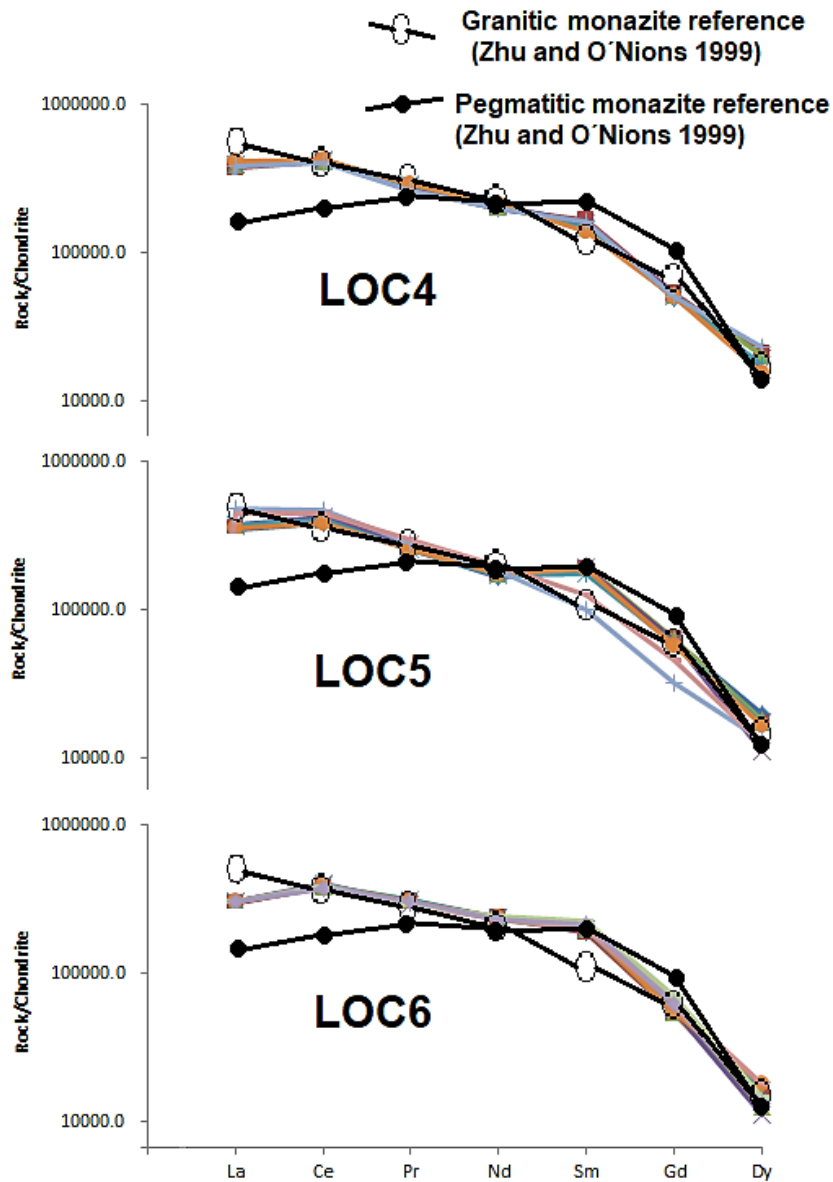
409

410 In all 3 sites, the sum of cations including P is 2, assuming an idealized monazite  
411 formula  $LnPO_4$ . Phosphor and oxygen deficits in all the formulas of the three sites  
412 are probably due to cation charge balance vacancies, especially of  $Ce^{(3+, 4+)}$ , and  
413 possible occupancy of elements like Si on the P-site.

414

415 Also, the chondrite-normalized REE distribution patterns in Figure 6 for the  
416 monazite crystals from LOC (4, 5 and 6) are very similar among each another.  
417 Their REE pattern is much closer to granitic than pegmatitic monazites as defined  
418 by Zhu and O'Nions (1999), especially exhibiting higher La and Ce contents than  
419 the pegmatitic reference monazites. The Gd and Dy depletion is characteristic of  
420 the monazite structure, and HREE enter preferentially in minerals like xenotime,  
421 as garnet is nearly absent.

422



424

425 Figure 6 Chondrite-normalized (Sun and McDonough, 1989) REE patterns of the  
 426 investigated monazites (Ho, Er, Tm, Yb and Lu below EPMA-detection limit) and  
 427 comparison with pegmatitic and granitic monazites after Zhu and O'Nions (1999). Observe  
 428 the higher coincidence of E-Colombian monazites with those of granitic origin with higher  
 429 La and Ce in comparison to Zhu and O'Nions (1999) pegmatitic monazites.

430

## 431 6.2 Geochronology

432



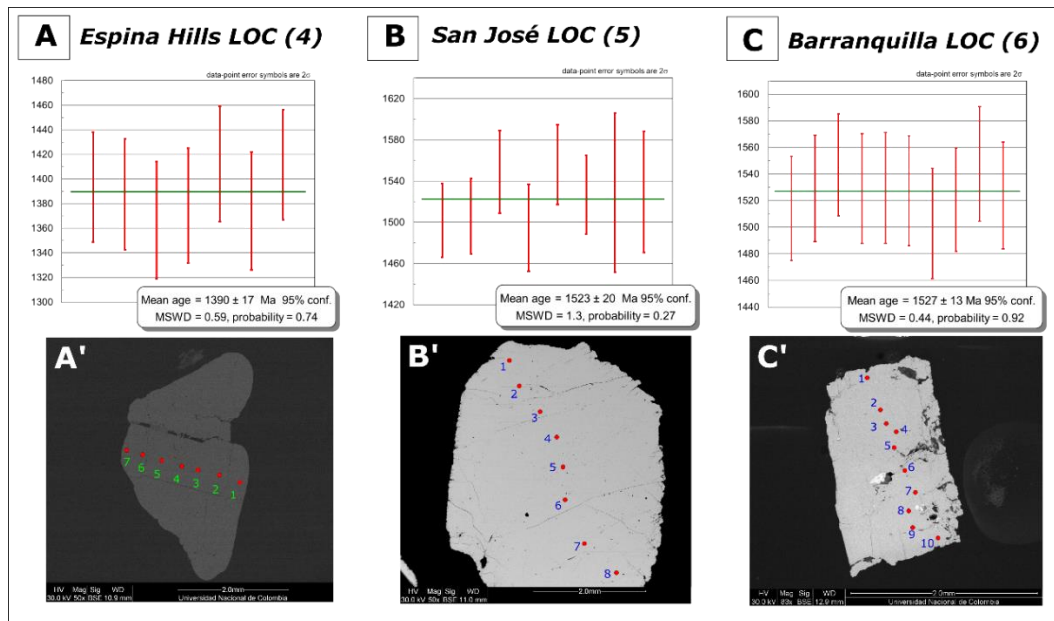
433

## 6.2.1 EPMA U-Th-Pb chemical ages

434

Th and Pb contents of all dated monazite grains are relatively high which helps to minimize background related errors (Jercinovic et al., 2008). Seven analyses of the monazite crystal from LOC (4) in Espina Hills yield an EPMA age of  $1390 \pm 17$  Ma ( $2\sigma$ , MSWD = 0.59) (Figure 7A). Eight analyses from the San Jose monazite crystal LOC (5) return a chemical age of  $1523 \pm 20$  Ma ( $2\sigma$ , MSWD = 1.3) (Figure 7B). Ten spots on a single monazite grain from Barranquilla LOC (6) yield a chemical age of  $1527 \pm 13$  Ma ( $2\sigma$ , MSWD = 0.27) (Figure 7C).

440



441

442 Figure 7. EPMA U-Th-Pb average ages calculated for each sample corresponding to  
443 Espina Hills (A), San José (B) and Barranquilla (C) localities. Data point errors are reported  
444 as  $2\sigma$ . Below, BSE images of the corresponding monazite crystals showing the analyzed  
445 points (A', B' and C'). Data table in Annex B.

446

## 6.2.2 LA-ICP-MS U-Th-Pb ages

447

The twenty-two analyses in 15 monazite grains from LOC (4) expressed as  $^{208}\text{Pb}/^{232}\text{Th}$  versus  $^{206}\text{Pb}/^{238}\text{U}$  concordia diagram (Figure 8A) plot all in a concordant position and yield a concordia age of  $1346 \pm 18$  Ma (MSWD (conc+equiv) = 1).

450

451

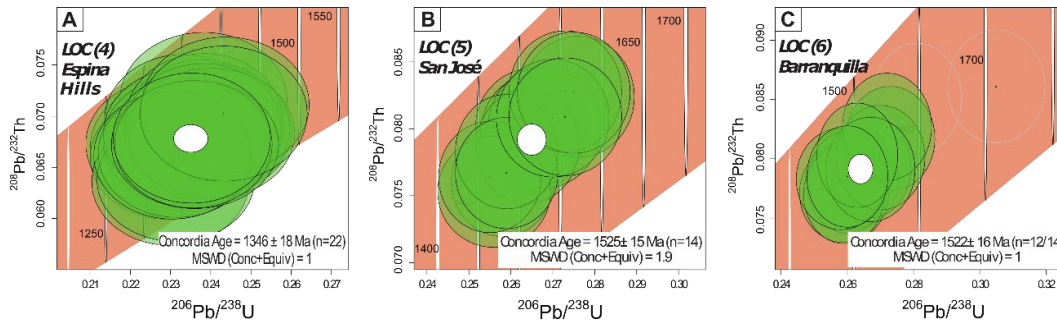
The fourteen analyses in 10 monazite grains of sample LOC (5) plot all in a concordant position (Figure 8B). Although they spread along the concordia curve with apparent ages from 1606 down to 1468 Ma, they yield a relatively well constrained concordia age of  $1525 \pm 15$  Ma (MSWD (conc+equiv) = 1.9).

454

455

The fourteen analyses in 5 monazite grains of sample LOC (6) plot in a concordant position (Figure 8C). Two analyses (grey lines in Figure 8C) yield apparent ages of 1605 and 1650 Ma, respectively. However, the twelve remaining analyses yield a well-constrained concordia age of  $1522 \pm 16$  Ma (MSWD (conc+equiv) = 1).

458



459

460 Figure 8. LA-ICP-MS monazite concordia ages of localities 4-6: Espina Hills (A), San José  
 461 (B) and Barranquilla (C). Data table in Annex C. Data errors are reported at  $2\sigma$ .

462

## 463 7 Discussion

### 464 7.1 Chemical data

465 The samples from this study were collected at the surface from colluvial deposits,  
 466 weathered zones, and rock fragments, without the possibility of studying the  
 467 magmatic to pegmatitic bodies present at depth. However, the geological,  
 468 mineralogical, and geochemical data allow us to draw some preliminary, although  
 469 contradictory conclusions about the three sites.

470 The chemical data suggest that the monazite crystals from all sites are very similar  
 471 despite their distance of 70-150 km among each another. They are consistent with  
 472 a granitic to pegmatitic origin, but without reaching highly specialized rare  
 473 elements pegmatite characteristics. Parts of the mineral assemblage are  
 474 indicative of a NYF-family affinity, with a geochemical signature  $Nb > Ta$ ,  $Ti$ ,  $Y$ ,  $Sc$ ,  
 475  $REE$ ,  $Zr$ ,  $U$ ,  $Th$ ,  $F$  (Černý and Ercit, 2005).

476 Melcher et al. (2017) proposed a possible NYF-family affinity for placer tapiolite,  
 477 ixiolite, CGM (columbite group minerals) and rutile samples from not specified E-  
 478 Colombian sites. One of their columbite, rutile and ixiolite samples however was  
 479 collected near Barranco Minas at the Guaviare River (Figure 2). The presence of  
 480 cassiterites and some personal information from the authors demonstrate that  
 481 most of these samples were collected within the Vichada Department associated  
 482 with the Parguaza Rapakivi Granite intrusion (see LOC (2) in Figure 2), which is  
 483 also in agreement with the higher Sn-contents reported by Melcher et al., (2017).  
 484 However, cassiterites were not found in panning concentrates in the Guainía  
 485 Department study area. In the ixiolite and CGM-samples those authors also  
 486 observed a CI-chondrite normalized HREE-enrichment trend and negative Eu and  
 487 Y anomalies (Melcher et al., (2017).

488

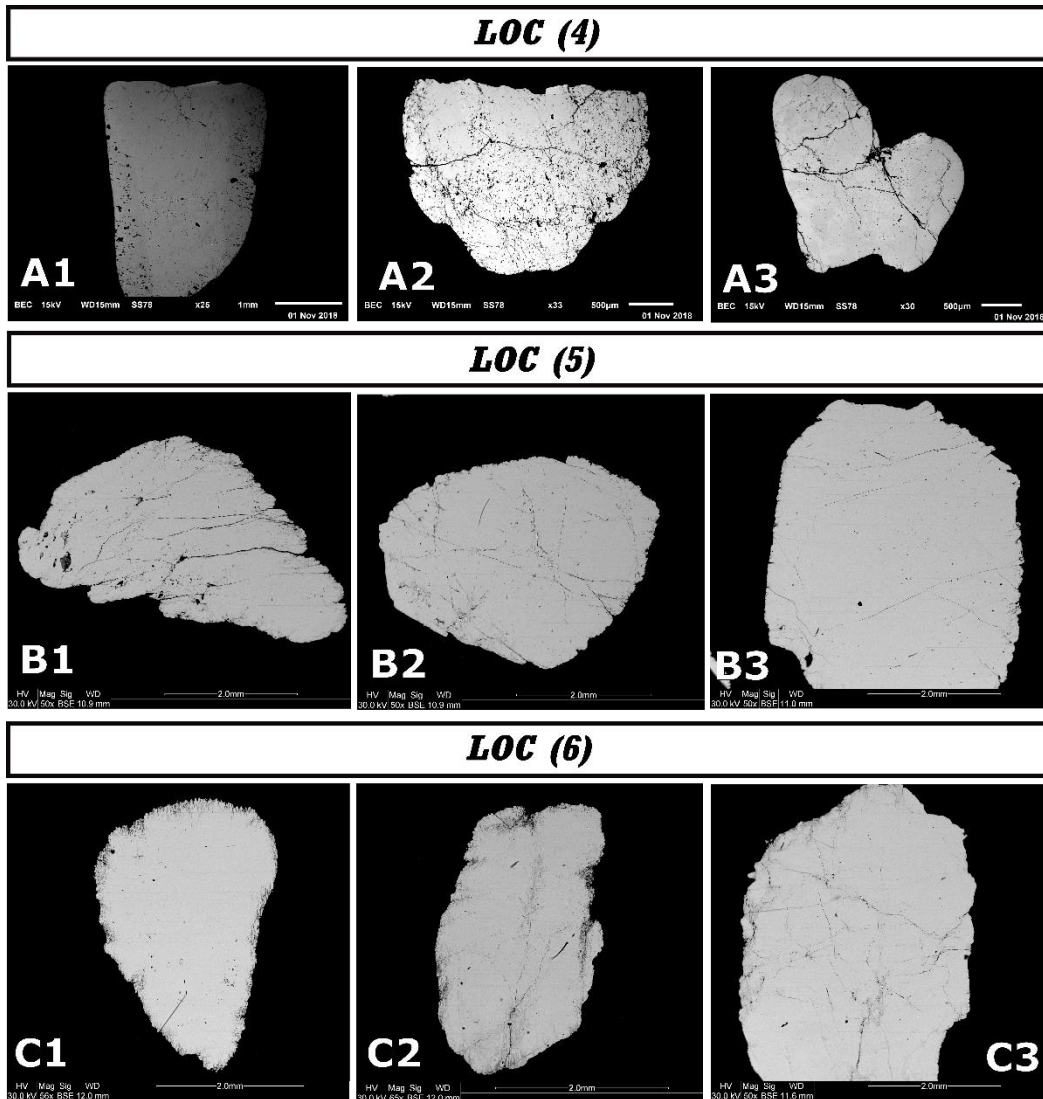
489 Chappell and White (2001) recognized based on whole rock geochemistry an  
 490 elevated phosphorus content as diagnostic of the S-type granites, and hence of  
 491 LCT-pegmatites, whereas the NYF pegmatites are depleted in phosphorus,

492 tourmaline is uncommon and abundant fluorite or topaz would reflect the  
493 enrichment in fluorine (Cerný et al., 2012). If this is true, the abundant monazite,  
494 xenotime, and black tourmaline concentrations in the three sampling sites as well  
495 as the absence of fluor-bearing minerals advocate against a NYF affinity. The  
496 REE-monazite data of Zhu and O’Nions (1999) suggest a more granitic than  
497 pegmatitic origin of the mineralizations but are only one indication. Also, as the  
498 samples are from colluvial deposits, it may be that the two families of pegmatites  
499 (LCT and NYF) have been eroded and contributed to the overall chemical and  
500 mineralogical composition of the studied samples. We can therefore at this stage  
501 conclude that the mineralogical and geochemical data are not very conclusive to  
502 propose a clear affinity but support a more complex genesis that involve different  
503 primary sources related with S- and A-type granites from which mineralized  
504 pegmatites may have evolved.

505

## 506 **7.2 Age and interpretations**

507 We used EPMA and LA-ICP-MS as complementary technique, EPMA in this study  
508 was use for acquiring the chemical composition of the monazite as well as for  
509 calculating the chemical age while LA-ICP-MS was used to measure the isotopes  
510 U-Th-Pb for calculating directly the ages in a faster way which is better to do  
511 provenance analysis. LOC (5 and 6) yield an EPMA chemical and LA-ICP-MS  
512 ages of  $1523 \pm 20$  Ma and  $1525 \pm 15$  Ma and  $1527 \pm 13$  Ma and  $1522 \pm 16$  Ma  
513 respectively, all within the error, however LOC (4) yield an EPMA chemical and  
514 LA-ICP-MS ages of  $1390 \pm 17$  Ma and  $1346 \pm 18$  Ma respectively, a difference of  
515  $\sim 45$  Ma was found. This difference may be attributed to EPMA measurements due  
516 to imperfections in polishing. BSE Images (Figure 9) revealed a homogeneous  
517 non-zoned without inclusion monazite crystals for the three localities. For the other  
518 hand xenotime from the same pan concentrate used for LOC (4) yield an LA-ICP-  
519 MS U-Pb age of  $1390 \pm 8$  Ma (unpublished data yet) that could be related.



520

521 Figure 9. BSE images for some crystals use for LA-ICP-MS dating for the three localities  
 522 showing a very homogeneous crystals of monazite. showing a rich thorium mineral were  
 523 observed; (A1, 2 and 3) Monazite crystals from LOC (4); (B1, 2 and 3) Monazite crystals  
 524 from LOC (5), small inclusion < 30  $\mu\text{m}$  of Th mineral was observed; (C1, 2 and 3) Monazite  
 525 crystals from LOC (6).

526

### 527 7.2.1 Age relation with the surrounding rocks

528 The oldest EPMA and LA-ICP-MS based ages at LOC (4)  $1390 \pm 17$  Ma and  $1346$   
 529  $\pm 18$  Ma, LOC (5)  $1523 \pm 20$  Ma and  $1525 \pm 15$  and LOC (6)  $1523 \pm 20$  Ma and  
 530  $1525 \pm 15$  Ma, respectively, indicate a crystallization age similar to  
 531 Mesoproterozoic anorogenic intrusions recorded in homogeneous anorogenic A-  
 532 type granites present in the study area (Table 1). This magmatic episodes  
 533 represented by anorogenic granites in the surroundings of the Orinoco, Guainía,  
 534 Inírida and Cuyari Rivers may be locally related with coeval granites from Vaupés  
 535 River and regionally with rapakivi granites of the Pijiguaos range and Parguaza in

536 Venezuela with reported ages of 1545-1500 Ma and in Brazil with granites from  
 537 the intrusive suite of Serra da Provincia with U-Pb ages of 1600-1532 Ma (see  
 538 Table 1), intruded at the end of the Rio Negro Juruena orogeny. Several of these  
 539 intrusive rocks and their post magmatic phases are the primary source of Sn, Ta,  
 540 Nb and Ti mineralizations.

541 The crystallization ages obtained from the monazite crystals from the three  
 542 localities are comparable with those from exposed Mesoproterozoic rocks such  
 543 as Parguaza in Venezuela and Vichada, Colombia from Orinoco River, Danta and  
 544 Matraca granites from the Inírida River, Tabaquen and similar granites from the  
 545 Guainía River and the rapakivi granites from the Cuyarí River reported in the  
 546 Guainía Department (see Figure 2 and Table 1)). The oldest obtained age range  
 547 of 1527-1525 Ma from the localities (5, 6) may correspond to monazite  
 548 mineralization from pegmatitic rocks related to granites near to Tabaquen and San  
 549 José with U-Pb ages of 1606-1525 Ma and rapakivi granites at Cuyarí River with  
 550 ages between 1550 -1600 Ma, and the younger ones in the locality (4) with an  
 551 age range of 1346 to 1390 Ma could correspond to monazite mineralization from  
 552 pegmatites of Espina, Danta and Matraca granitic rocks, the last one with an age  
 553 of ~1343 Ma (Table 1).

554

555 Table 1. Compiled ages of some Mesoproterozoic granites from Colombia, Venezuela,  
 556 and Brazil including the isotopic method use to dating

Locality	Country	Isotopic system	Age (Ma)	Reference
Murciélago Hill (Rapakivi granite)	Colombia	Ar/Ar	1237 ± 13	SGC (2013)
Murciélago Hill (Rapakivi granite)	Colombia	Ar/Ar	1292 ± 8	SGC (2013)
Angelita Hill (Rapakivi granite)	Colombia	Ar/Ar	1359 ± 8	SGC (2013)
Bitá Hill (Rapakivi granite)	Colombia	Ar/Ar	1361 ± 9	SGC (2013)
La Hormiga Hill (Rapakivi granite)	Colombia	Ar/Ar	1359 ± 8	SGC (2013)
La Hormiga Hill (Rapakivi granite)	Colombia	Ar/Ar	1339 ± 8	SGC (2013)
La Hormiga Hill (Rapakivi granite)	Colombia	Ar/Ar	1366 ± 9	SGC (2013)
La Hormiga Hill (Rapakivi granite)	Colombia	Ar/Ar	1366 ± 9	SGC (2013)
La Hormiga Hill (Rapakivi granite)	Colombia	Ar/Ar	1376 ± 14	SGC (2013)
La Hormiga Hill (Rapakivi granite)	Colombia	Ar/Ar	1383 ± 8	SGC (2013)
La Venturosa (Rapakivi granite)	Colombia	Ar/Ar	1368 ± 14	SGC (2013)
La Venturosa (Rapakivi granite)	Colombia	Ar/Ar	1361 ± 8	SGC (2013)
Tomo River (Rapakivi granite)	Colombia	U/Pb	1392 ± 5	Bonilla et al. (2013)
Cachicamo Hills (Rapakivi granite)	Colombia	U/Pb	1402 ± 2	Bonilla et al. (2013)
Inírida River, Matraca and Danta rapakivi granites	Colombia	U/Pb	1343 ± 8	Bonilla et al. (2016)

Locality	Country	Isotopic system	Age (Ma)	Reference
Apaporis River (Syenogranite)	Colombia	U/Pb	1593 ± 06	Ibañez-Mejia et al. (2011)
Apaporis River (Syenogranite)	Colombia	U/Pb	1578 ± 27	Ibañez-Mejia et al. (2011)
Apaporis River (Monzogranite)	Colombia	U/Pb	1530 ± 21	Ibañez-Mejia et al. (2011)
Vaupés River (monzogranite)	Colombia	U/Pb	1574 ± 10	Ibañez-Mejia et al. (2011)
Mitú granite	Colombia	U/Pb	1552 ± 34	Priem et al. (1982)
Raudal Morroco Inírida River (Monzogranite)	Colombia	U/Pb	1507 ± 22	Cordani et al. (2016)
Mitú range (Monzogranite)	Colombia	U/Pb	1510 ± 26	Cordani et al. (2016)
Matraca granite - Inírida River	Colombia	U/Pb	1343 ± 8	Bonilla et al. (2016)
Granites of the Guainía River	Colombia	U/Pb	1606 ± 46	Bonilla et al. (2019)
Granites of the Guainía River	Colombia	U/Pb	1525 ± 27	Bonilla et al. (2019)
Cuyari River (Porphyritic granite)	Colombia	U/Pb	1596 ± 52	Bonilla et al. (2019)
Marieta River, Marieta Granite	Venezuela	Rb/Sr	1340 ± 10	Cited in Barrios et al. (1985)
San Pedro Granite	Venezuela	Rb/Sr	1372 ± 10	Cited in Barrios et al. (1985)
Parguaza Granite	Venezuela	Rb/Sr	1386 ± 28	Cited in Barrios et al. (1985)
Parguaza Granite	Venezuela	Rb/Sr	1486 ± 25	Bogotá (1981)
Los Pijiguaos Granite	Venezuela	Rb/Sr	1531 ± 39	Gaudette et al. (1978)
Parguaza Granite	Venezuela	Rb/Sr	1531 ± 39	Bogotá (1981)
Marieta Granite	Venezuela	Rb/Sr	1534 ± 13	Barrios y Rivas (1980)
Parguaza Granite	Venezuela	U/Pb	1545 ± 20	Gaudette et al. (1978)
Intrusive suite Alto Candeias	Brazil	U/Pb	1338-1346	Bettencourt et al. (1999)
Intrusive suite Teotônio	Brazil	U/Pb	1387 ± 16	Bettencourt et al. (1999)
Intrusive suite Santo Antônio	Brazil	U/Pb	1406 ± 32	Bettencourt et al. (1999)
Mucajaí Granite	Brazil	U/Pb	1544 ± 42	Gaudette et al. (1996)
Içana River (two- mica granite)	Brazil	Pb/Pb	1521 ± 32	Almeida et al. (1997)
Papuri River granite	Brazil	U/Pb	1521 ± 13	Tassinari et al. (1996)
Charnockite Serra da Prata	Brazil	U/Pb	1564 ± 21	Fraga et al. (1997)
Surucucú Granite	Brazil	Rb/Sr	1583	Dall'Agnol et al. (1975)
Intrusive suite Serra da Providencia	Brazil	U/Pb	1606 -1532	Bettencourt et al. (1999)

557

## 558 7.2.2 Age relations with Nb-Ta, U-Th bearing minerals

559 Melcher et al., (2017) based on U–Pb isotopes in wodginite–ixiolite, CGM, rutile  
560 and cassiterite, estimated a mineralization age ranging from 1453 to 1277 Ma for  
561 their Colombian samples, although they did not report their exact provenances.

562 For other hand samples of LOC (2) and Venezuela were mineralogically  
563 compared and rutile-(Nb,Ta) U-Pb ages are  $1512 \pm 12$  Ma (Franco et al., 2021).

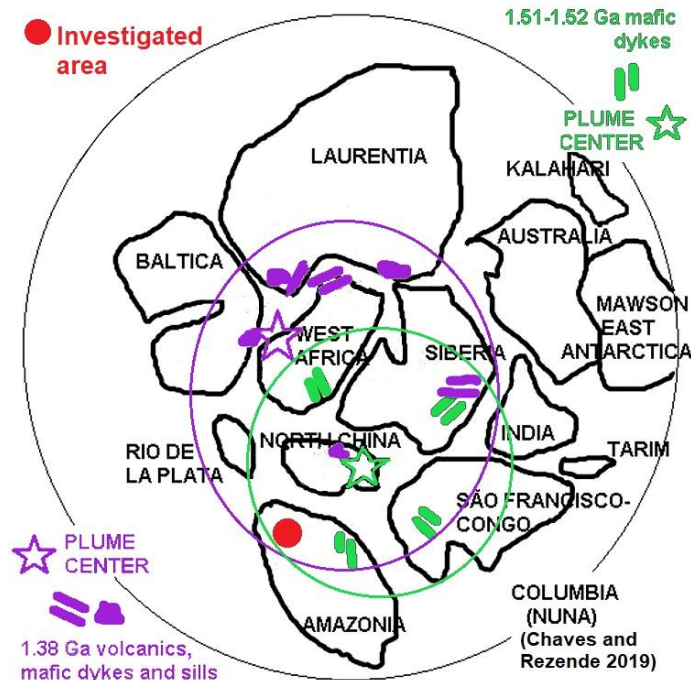
### 564 **7.3 Intraplate tectono-magmatic phenomena and paleogeographic** 565 **implications**

566 Intraplate tectono-magmatic phenomena are generally attributed to the ascent of  
567 deep mantle plumes, arising from the core–mantle boundary. Mantle plumes are  
568 consistent with experimental studies and mantle seismic tomography. In addition,  
569 the assembly of supercontinents and their subsequent breakup are inferred to  
570 relate to the arrival of mantle plumes (Ernst, 2014). The rising of mantle plumes  
571 and/or asthenospheric melts results in tectonic and magmatic manifestations that  
572 include crustal uplifts, continental rifting, the emplacement of anorogenic  
573 magmas, and LIPs (large igneous provinces) on continents. Some of the most  
574 significant global anorogenic magmatic rocks of Earth's history are recognized in  
575 the period between 1300 and 1800 Ma (Ernst, 2014). For comparison, in  
576 Laurentia, a distinctive A-type or “anorogenic” granites of  $\sim 1.4$  Ga age was  
577 emplaced heterogeneous Proterozoic crust see Goodge and Vervoort (2006),  
578 globally this magmatism according to conclusion in (Vigneresse, 2005) appears to  
579 be unique because it requires a supercontinent with a zone of juvenile crust  
580 surrounded by older cratons.

581 Huge Fe-Ti-V deposits hosted by anorogenic gabbro-anorthosite complexes, and  
582 A-type granite related systems enriched in elements such as Sn, W, Cu, Zn, Bi,  
583 Mo, U, and F are related to intraplate processes and mantle plume associated  
584 magmatism (Pirajno, 2007).

585 Based on the distribution of fragments of LIP of distinct ages, Chaves and  
586 Rezende (2019) have proposed a new reconstruction of Columbia (Nuna)  
587 supercontinent. This reconstruction is shown in Figure 10, where purple and green  
588 circles represent the thermal impact area promoted by 1.52 Ga and 1.38 Ga  
589 mantle plume activity, recorded by volcanic mafic dykes and sills (LIP fragments).  
590 The red circle in (Figure 10) corresponds to the studied area in the NW Amazon  
591 Craton, which probably suffered partial melting generating A-type granites and  
592 associated pegmatites with an age comparable to the one of mantle plumes.

593



594

595 Figure 10. Columbia (Nuna) supercontinent reconstructed after Chaves and Rezende  
 596 (2019). Purple and green circles represent the thermal impact area promoted by 1.52 Ga  
 597 and 1.38 Ga mantle plume activity, recorded by their associated LIP fragments (volcanic,  
 598 mafic dykes and sills). The red dot corresponds to the study area in the NW-Amazon  
 599 Craton.

600

## 601 8 Conclusions

602 The monazite ages obtained by EPMA and LA-ICP-MS dating are in the same  
 603 range for LOC (5) and (6) confirm that EPMA may be a useful tool to establish the  
 604 crystallization age of monazite crystals poor in initial Pb. The high U, Th and  
 605 radiogenic Pb contents and lack of inclusions and zonation of the homogeneous  
 606 monazite crystals may make them suitable as internal standard material, however  
 607 it would be necessary to find their primary source rocks, since there are age  
 608 variations between the samples and the monazite grains from various primary  
 609 sources may be in the same colluvial deposit.

610 The two age ranges suggest at least two enrichment events related with felsic  
 611 intrusions and possibly associated pegmatites. The oldest ca. 1527 Ma processes  
 612 forming CGM and the youngest of ca. 1346-1390 Ma forming REE and U-Th  
 613 bearing minerals and in low proportion rutile-(Nb,Ta) and CGM.

614 The several kinds of ore minerals found in the three localities suggest different  
 615 enrichment cycles linked to Mesoproterozoic magmatism. The Nb, Ta, U, Th and  
 616 REE mineralizations may be result of pegmatitic phases of 1.52 Ga and 1.38 Ga  
 617 anorogenic granitic magmatism triggered by mantle plumes affecting the  
 618 Columbia (Nuna) supercontinent.



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## 11 Annex, supplementary tables

Annex A. Supplementary table of operating conditions for the LA-ICP-MS equipment (GeOHeLiS Platform).

### Laboratory & Sample Preparation

Laboratory name	GeOHeLiS Platform, University of Rennes, Rennes, France
Sample type/mineral	Monazite
Sample preparation	Separated grain mounted in epoxy pucks

### Laser ablation system

Make, Model & type	ESI NWR193UC, Excimer
Ablation cell	ESI NWR TwoVol2
Laser wavelength	193 nm
Pulse width	< 5 ns
Fluence	5 J/cm <sup>2</sup>
Repetition rate	3Hz
Spot size	8 µm
Sampling mode / pattern	Single spot
Carrier gas	100% He, Ar make-up gas and N <sub>2</sub> (3 ml/mn) combined using in-house smoothing device
Background collection	20 seconds
Ablation duration	60 seconds
Wash-out delay	15 seconds
Cell carrier gas flow (He)	0.75 l/min

### ICP-MS Instrument

Make, Model & type	Agilent 7700x, Q-ICP-MS
Sample introduction	Via conventional tubing
RF power	1350W
Sampler, skimmer cones	Ni
Extraction lenses	X type
Make-up gas flow (Ar)	0.85 l/min
Detection system	Single collector secondary electron multiplier
Data acquisition protocol	Time-resolved analysis
Scanning mode	Peak hopping, one point per peak
Detector mode	Pulse counting, dead time correction applied, and analog mode when signal intensity > ~ 10 <sup>6</sup> cps
Masses measured	<sup>204</sup> (Hg + Pb), <sup>206</sup> Pb, <sup>207</sup> Pb, <sup>208</sup> Pb, <sup>232</sup> Th, <sup>238</sup> U
Integration time per peak	10-30 ms
Sensitivity / Efficiency	22000 cps/ppm Pb (50µm, 10Hz)

### Data Processing

Gas blank	20 seconds on-peak
Calibration strategy	Moacir monazite standard used as primary reference material, Managoutry monazite used as secondary reference material (quality control)

Reference Material info	Moacir (Gasquet et al., 2010) Manangoutry (Paquette and Tiepolo, 2007)
Data processing package used	GLITTER <sup>®</sup> (van Achterbergh et al., 2001)
Mass discrimination	Standard-sample bracketing with <sup>207</sup> Pb/ <sup>206</sup> Pb, <sup>206</sup> / <sup>238</sup> U, <sup>208</sup> Pb/ <sup>232</sup> Th normalized to reference material Moacir
Common Pb correction	No common Pb correction
Uncertainty level and propagation	Ages are quoted at 2 sigma absolute, propagation is by quadratic addition according to Horstwood et al. (2016). Reproducibility and age uncertainty of reference material are propagated.
Quality control / Validation	Manangoutry : 548 ± 6 Ma (MSWD=0.98 ; n=11)

Annex B. EPMA mineral chemistry data of monazite crystals from the localities Espina Hills LOC (4); San José LOC (5) and Barranquilla LOC (6) and ages calculated using Th, U and Pb contents. All elements are expressed as main oxides measure in weighted percent (wt.%) independently of their real oxidation state.

Sample	Analysis point/ oxide	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	ThO <sub>2</sub>	UO <sub>2</sub>	Y <sub>2</sub> O <sub>3</sub>	La <sub>2</sub> O <sub>3</sub>	Ce <sub>2</sub> O <sub>3</sub>	Pr <sub>2</sub> O <sub>3</sub>	Nd <sub>2</sub> O <sub>3</sub>	Sm <sub>2</sub> O <sub>3</sub>	Gd <sub>2</sub> O <sub>3</sub>	Dy <sub>2</sub> O <sub>3</sub>	CaO	PbO	Total	U (ppm)	2σ %	Th (ppm)	2σ %	Pb (ppm)	2σ %	Age (Ma)	2σ (abs)
LOC 4	1	27.99	1.05	8.65	0.27	1.38	10.83	30.00	3.04	10.75	2.90	1.18	0.64	1.49	0.57	100.73	1896	5.3	76016	1	5291	1.9	<b>1393</b>	45
	2	26.28	1.07	8.56	0.26	1.51	10.54	29.74	3.02	11.02	2.97	1.28	0.61	1.46	0.56	98.86	1813	5.5	75225	1	5198	1.9	<b>1387</b>	45
	3	26.32	1.16	8.14	0.18	1.45	11.00	30.23	3.04	11.27	2.77	1.23	0.60	1.13	0.51	99.02	1131	8.8	71534	1	4734	2.1	<b>1366</b>	48
	4	27.30	1.14	8.33	0.20	1.40	11.10	30.48	3.16	11.31	2.83	1.28	0.47	1.22	0.53	100.73	1297	7.7	73204	1	4920	2.0	<b>1378</b>	47
	5	26.08	1.37	8.51	0.18	1.41	11.00	30.23	3.25	11.52	2.60	1.19	0.51	0.94	0.55	99.32	1111	9.0	74786	1	5106	2.0	<b>1412</b>	47
	6	25.55	1.62	8.09	0.18	1.29	11.46	30.74	3.23	11.81	2.49	1.21	0.45	0.80	0.51	99.42	1134	8.8	71095	1	4734	2.1	<b>1374</b>	48
	7	26.15	1.04	8.65	0.28	1.48	10.71	29.31	2.96	10.86	2.89	1.21	0.68	1.45	0.58	98.23	1984	5.0	76016	1	5384	1.9	<b>1412</b>	45
LOC 5	1	26.26	1.26	12.23	0.46	1.53	9.63	27.55	2.86	9.16	3.26	1.49	0.58	2.02	0.89	99.18	3371	3.0	107477	1	8262	1.2	<b>1502</b>	36
	2	27.61	1.19	11.95	0.44	1.22	9.95	28.21	2.95	9.49	3.40	1.52	0.50	1.91	0.87	101.21	3210	3.1	105017	1	8076	1.2	<b>1506</b>	37
	3	25.88	1.15	10.49	0.40	1.29	10.10	28.86	3.01	9.56	3.53	1.53	0.53	1.65	0.79	98.75	2939	3.4	92186	1	7334	1.4	<b>1549</b>	40
	4	26.40	1.06	9.56	0.35	0.98	10.49	30.38	3.08	9.93	3.48	1.46	0.32	1.52	0.69	99.69	2551	3.9	84013	1	6405	1.6	<b>1495</b>	42
	5	25.10	1.23	11.04	0.44	1.14	10.42	29.41	2.87	9.38	3.09	1.40	0.50	1.78	0.84	98.64	3261	3.1	97020	1	7798	1.3	<b>1556</b>	39
	6	25.39	1.20	11.14	0.44	1.20	9.83	27.86	2.88	9.66	3.34	1.36	0.49	1.85	0.83	97.47	3255	3.1	97898	1	7705	1.3	<b>1527</b>	38
	7	27.96	0.77	4.10	0.21	1.44	13.58	33.57	3.15	10.08	1.79	0.77	0.39	0.59	0.32	98.71	1578	6.3	36004	1.00	2933	3.41	<b>1529</b>	77
	8	27.49	1.34	5.86	0.27	1.55	12.35	32.03	3.31	10.95	2.22	1.10	0.37	0.72	0.45	100.00	2026	4.9	51506	1.00	4140	2.42	<b>1530</b>	59
LOC 6	1	23.43	2.27	10.89	0.37	2.11	8.36	27.95	3.43	12.98	3.34	1.39	0.43	0.73	0.79	98.46	2652	3.8	95701	1	7334	1.4	<b>1514</b>	39
	2	24.17	2.20	10.72	0.34	2.15	8.39	27.91	3.41	12.85	3.40	1.28	0.44	0.73	0.78	98.77	2397	4.2	94207	1	7241	1.4	<b>1529</b>	40
	3	24.88	2.44	11.49	0.38	2.18	8.46	27.97	3.47	12.72	3.51	1.31	0.37	0.72	0.85	100.73	2707	3.7	100974	1	7891	1.3	<b>1547</b>	38
	4	24.27	2.08	10.07	0.35	1.94	8.60	28.78	3.22	13.15	3.57	1.33	0.32	0.69	0.74	99.10	2522	4.0	88495	1	6869	1.5	<b>1529</b>	41
	5	24.89	2.05	9.95	0.34	2.02	8.54	28.28	3.48	13.13	3.56	1.36	0.47	0.71	0.73	99.51	2441	4.1	87441	1	6777	1.5	<b>1530</b>	42
	6	25.27	2.12	10.12	0.34	2.12	8.65	28.19	3.38	13.02	3.66	1.31	0.52	0.68	0.74	100.11	2431	4.1	88935	1	6869	1.5	<b>1527</b>	41
	7	25.30	1.50	9.81	0.35	2.19	8.51	28.21	3.37	12.54	3.69	1.59	0.41	1.10	0.71	99.29	2537	3.9	86210	1	6591	1.5	<b>1503</b>	42

S a m p l e	Analysis point/ oxide	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	ThO <sub>2</sub>	UO <sub>2</sub>	Y <sub>2</sub> O <sub>3</sub>	La <sub>2</sub> O <sub>3</sub>	Ce <sub>2</sub> O <sub>3</sub>	Pr <sub>2</sub> O <sub>3</sub>	Nd <sub>2</sub> O <sub>3</sub>	Sm <sub>2</sub> O <sub>3</sub>	Gd <sub>2</sub> O <sub>3</sub>	Dy <sub>2</sub> O <sub>3</sub>	CaO	PbO	Total	U (ppm)	2σ %	Th (ppm)	2σ %	Pb (ppm)	2σ %	Age (Ma)	2σ (abs)
	8	24.89	1.87	11.07	0.39	2.06	8.56	26.92	3.22	12.71	3.55	1.50	0.52	1.17	0.81	99.24	2819	3.5	97283	1	7519	1.3	1521	39
	9	24.74	1.53	9.62	0.31	1.90	8.59	27.62	3.26	13.33	4.00	1.68	0.43	1.08	0.71	98.80	2195	4.6	84541	1	6591	1.5	1548	43
	10	25.66	1.76	10.61	0.34	2.08	8.55	27.08	3.39	12.77	3.82	1.46	0.39	1.27	0.77	99.95	2404	4.2	93241	1	7148	1.4	1524	40

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2 Annex C. LA-ICP-MS U/Th/Pb isotopic data of monazite crystals from Espina Hills LOC (4), San José LOC (5) and Barranquilla LOC (6).

Points of ablation	<sup>208</sup> Pb/ <sup>232</sup> Th	PropErr2σAbs	<sup>206</sup> Pb/ <sup>238</sup> U	PropErr2σAbs	Age <sup>206</sup> Pb/ <sup>238</sup> U	PropErr2σAbs	Age <sup>208</sup> Pb/ <sup>232</sup> Th	PropErr2σAbs
Mnz_Loc (4) p1	0.0670	0.0051	0.230	0.016	1327	22	1311	14
Mnz_Loc (4) p2	0.0660	0.0050	0.230	0.016	1327	26	1291	16
Mnz_Loc (4) p3	0.0716	0.0054	0.238	0.017	1377	22	1397	17
Mnz_Loc (4) p4	0.0685	0.0052	0.239	0.017	1380	19	1339	15
Mnz_Loc (4) p5	0.0673	0.0051	0.229	0.016	1322	22	1316	16
Mnz_Loc (4) p6	0.0697	0.0052	0.233	0.016	1352	29	1361	17
Mnz_Loc (4) p7	0.0690	0.0053	0.238	0.016	1371	22	1350	16
Mnz_Loc (4) p8	0.0687	0.0053	0.239	0.017	1379	22	1343	16
Mnz_Loc (4) p9	0.0636	0.0051	0.230	0.016	1325	55	1245	26
Mnz_Loc (4) p10	0.0676	0.0052	0.233	0.016	1347	30	1321	19
Mnz_Loc (4) p11	0.0637	0.0049	0.231	0.016	1334	28	1248	15
Mnz_Loc (4) p12	0.0683	0.0052	0.233	0.016	1345	27	1334	20
Mnz_Loc (4) p13	0.0664	0.0051	0.232	0.016	1340	23	1298	16
Mnz_Loc (4) p14	0.0696	0.0053	0.238	0.016	1374	22	1360	16
Mnz_Loc (4) p15	0.0686	0.0053	0.238	0.016	1373	23	1341	18
Mnz_Loc (4) p16	0.0683	0.0052	0.240	0.017	1383	22	1335	16
Mnz_Loc (4) p17	0.0711	0.0054	0.244	0.017	1406	21	1388	16
Mnz_Loc (4) p18	0.0662	0.0051	0.231	0.016	1331	26	1294	17
Mnz_Loc (4) p19	0.0672	0.0051	0.236	0.017	1356	30	1314	16

Mnz_Loc (4) p20	0.0673	0.0051	0.236	0.016	1360	20	1315	15
Mnz_Loc (4) p21	0.0703	0.0054	0.243	0.017	1399	20	1371	16
Mnz_Loc (4) p22	0.0679	0.0052	0.235	0.016	1361	19	1326	13
Mnz_Loc (5) p1	0.0801	0.0036	0.265	0.011	1511	55	1556	67
Mnz_Loc (5) p2	0.0796	0.0036	0.265	0.010	1513	53	1547	67
Mnz_Loc (5) p3	0.0824	0.0037	0.276	0.011	1569	57	1599	70
Mnz_Loc (5) p4	0.0801	0.0037	0.268	0.011	1523	56	1556	69
Mnz_Loc (5) p5	0.0761	0.0034	0.258	0.010	1474	53	1482	64
Mnz_Loc (5) p6	0.0787	0.0035	0.264	0.010	1505	53	1530	66
Mnz_Loc (5) p7	0.0753	0.0034	0.256	0.011	1465	54	1468	63
Mnz_Loc (5) p8	0.0764	0.0034	0.255	0.010	1461	52	1488	64
Mnz_Loc (5) p9	0.0783	0.0035	0.259	0.010	1482	53	1523	65
Mnz_Loc (5) p10	0.0767	0.0034	0.259	0.010	1481	51	1492	64
Mnz_Loc (5) p11	0.0828	0.0037	0.273	0.011	1553	58	1606	69
Mnz_Loc (5) p12	0.0808	0.0036	0.270	0.011	1540	55	1570	67
Mnz_Loc (5) p13	0.0827	0.0037	0.275	0.011	1560	58	1606	69
Mnz_Loc (5) p14	0.0809	0.0036	0.273	0.011	1549	55	1571	67
Mnz_Loc (6) p1	0.0784	0.0035	0.262	0.011	1499	56	1525	66
Mnz_Loc (6) p2	0.0780	0.0035	0.260	0.010	1488	53	1517	65
Mnz_Loc (6) p3	0.0771	0.0034	0.259	0.011	1481	55	1500	64
Mnz_Loc (6) p4	0.0851	0.0038	0.281	0.011	1596	57	1649	70
Mnz_Loc (6) p5	0.0817	0.0036	0.273	0.011	1554	55	1587	68
Mnz_Loc (6) p6	0.0793	0.0035	0.267	0.010	1522	53	1543	66
Mnz_Loc (6) p7	0.0770	0.0034	0.259	0.010	1482	53	1499	64
Mnz_Loc (6) p8	0.0861	0.0039	0.305	0.012	1710	60	1669	72
Mnz_Loc (6) p9	0.0783	0.0035	0.258	0.011	1477	56	1522	66
Mnz_Loc (6) p10	0.0801	0.0036	0.270	0.011	1534	54	1557	67
Mnz_Loc (6) p11	0.0827	0.0037	0.272	0.011	1545	55	1605	69
Mnz_Loc (6) p12	0.0791	0.0036	0.267	0.011	1520	55	1538	67
Mnz_Loc (6) p13	0.0803	0.0036	0.263	0.010	1500	52	1560	67
Mnz_Loc (6) p14	0.0785	0.0035	0.260	0.010	1485	52	1527	65

