

**NIGERITE GROUP “ZINCONIGERITE-(6N6S)” VARIETY
IN THE RONCADEIRA PEGMATITE, NOVA PALMEIRA-COUNTY STATE
OF PARAÍBA, BORBOREMA PROVINCE, NE-BRAZIL**

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RESUMO

Descreve-se a primeira ocorrência de um mineral do grupo da nigerita na Província Pegmatítica da Borborema, no pegmatito Roncadeira, Nova Palmeira-PB. No Brasil é a segunda, pois já havia sido descrita outra, em regolito associado a pegmatitos próximos à vila Jornal, às margens do rio Parapueri, no Amapá. As análises químicas desta primeira ocorrência no Amapá não permitiram, na época, caracterizá-la conforme a nomenclatura adotada a partir de 2002 pela “International Mineralogical Association” (IMA) para o super-grupo nigerita-högbomita. A ocorrência de Roncadeira já havia sido mencionada em trabalho anterior pelos autores, mas sem maiores detalhes. A identificação foi realizada pela própria composição química, inicialmente semi-quantitativa, indicada por microscopia eletrônica de varredura (MEV), mas depois confirmada por análises quantitativas via microsonda eletrônica (EMPA) e pelo espectro RAMAN. A fórmula química obtida a partir da média de 13 análises em três grãos, normatizada para um total de 11 cátions é:



As médias das razões catiônicas $\text{Zn}/(\text{Zn}+\text{Fe}+\text{Mn}+\text{Mg}+\text{Ca}) = 0.56$ e de $\text{Zn}/\text{Fe} = 1.65$ (inadequadas para ferro- ou magnesianigerita) e a razão $\text{Al}/\text{Sn} = 8.27$ (maior que 7), típica para o polissoma -6N6S, permitem pois identificá-la como “zinconigerita-6N6S” (ZN a partir de agora), espécie do grupo ainda não reconhecida pela (IMA), mas já registrada em pelo menos oito outras ocorrências mundiais. A ZN no pegmatito de Roncadeira ocorre associada com cassiterita, tapiolita-(Fe), ferrowodginita, assim como com crisoberilo e gahnita enriquecidos tanto no contato “greisenificado” como em corpos de substituição dominados por agregados sacaroidais de albita + quartzo + muscovita. Esta associação é muito similar às descritas na ocorrência tipo na Nigéria e outras similares na Namíbia, Portugal (Cabana / Ponte de Lima; Lousas/Barroso Alvão) Amapá/Brasil, Finlândia (Rosendal). Todas estas ocorrências são, portanto possivelmente ligadas a atividades metamórfico-metassomáticas de contato tardias nos pegmatitos hospedeiros.

Palavras chave: “Zinconigerita-6N6S”, Pegmatito Roncadeira, Província Borborema.

ABSTRACT

A first occurrence of a nigerite-group mineral in the Borborema Pegmatite Province, at the Roncadeira pegmatite, Nova Palmeira county, State of Paraíba is described. It is the second one in Brazil after the first finding in the State of Amapá, in the regolith covering pegmatites close to the Jornal Village at the Parapuerí river, south of the Serra do Navio Mn-deposit. The specimens of the Amapá occurrences had been identified “sensu lato” as nigerite by wet chemical analyses without specification according to the nomenclature approved much later by the IMA (International Mineral Association) for the nigerite-högbomite supergroup. The Roncadeira occurrence was already mentioned earlier by the authors without details and identification embased on the chemical composition obtained by semi-quantitative SEM (scanning electron microscopy) followed by electron microprobe analyses (EMPA) and RAMAN spectra. The stoychiometric formula obtained by the average of thirteen EMPA data (on three grains), normalized for a total of 11 cations is



The average of the cationic ratios $\text{Zn}/(\text{Zn}+\text{Fe}+\text{Mn}+\text{Mg}+\text{Ca}) = 0.56$, and $\text{Zn}/\text{Fe} = 1.65$ are not compatible with ferro- or magnesian nigerite and $\text{Al}/\text{Sn} = 8.27$ (higher than 7.0) is typical for the 6N6S polysome, inadequate for the 2N1S polysome. These values would therefore allow to classify the nigerite group mineral from Roncadeira as “zinconigerite-6N6S” still not recognized as a valid species by IMA, but with similar chemistry found in other eight occurrences worldwide. The “zinconigerite” from Roncadeira occurs in paragenetic association with cassiterite, tapiolite-(Fe), ferrowodginite, ferrotitanowodginite and gahnite in both, the greisen-like border zone and in albite-rich replacement bodies in the inner pegmatite zones. In sheared portions this paragenesis is associated also with crysoberyl and sillimanite, features very similar to those registered at the type-locality in Nigeria and in Namibia, Portugal, Brazil (Amapá), Canada, Finland (Rosendal). All these occurrences are therefore possibly related with late contact-metamorphic-metassomatic processes of the host pegmatites.

Keywords: “Zinconigerite-6N6S”, Roncadeira Pegmatite, Província Borborema-NE-Brazil.

INTRODUCTION

Nigerite: from mineral to polytypes, polysomatic series and group.

For an easier understanding of what are the mineral species of the, today, nigerite-group it is appropriate to make a short historical report of the evolution toward the now accepted IMA-nomenclature. “Nigerite” was identified as a new rare tin-mineral species hosted in a pegmatite (at Egbe/Kabba) in Nigeria (Jacobson & Webb 1947 and Bannister *et al.*, 1947). Other occurrences of the mineral were discovered in Portugal (van Tassel

1965), Amapá, Brasil (Kloosterman 1974), Rosendal, Finland (Burke *et al.* 1977), Namíbia (Knorring & Condcliffe 1985), always in pegmatites, or in a peraluminous granite, as in Bohemia-Czech Republic (Čech *et al.* 1978). Later it was recognized to belong to a polysomatic series (Grey *et al.* 1979, Arakcheeva *et al.* 1995) forming the högbomite-nigerite supergroup, including the nigerite and högbomite groups (högbomite had been identified by Gavelin 1916). Later the taaffeite-group minerals were also included in the supergroup. The nomenclature and classification of the supergroup accepted by the International

Mineralogical Association (IMA) was explained in detail by Armbruster (2002) with the nigerite and högbomite groups as composed by modules of spinel- (S) ($=\text{T}_2\text{M}_4\text{O}_8$) and nolanite- (N) ($=\text{TM}_4\text{O}_7(\text{OH})$), piled along the crystallographic C-axis in variably ordered alternance and variable module compositions.

The distinction between the nigerite- and högbomite-groups is made according to the dominance of respectively Sn or Ti in the octahedral M-site of the nolanite module. Sub-groups are distinguished according to the composition of the tetrahedral T-site in the spinel module usually dominated either by Fe, Mg, Zn (respectively distinguished in the mineral name by the prefix ferro-, magnesio- or zinco). The mineral species in the sub-groups are distinguished, by the number and stacking alternance of the modules in the unit cell, identified by a suffix like 6N6S and -2N1S. In the case of the nigerite group IMA-accepted species are ferronigerite-2N1S and -6N6S, and magnesonigerite-2N1S and -6N6S.

In the högbomite group the procedure for mineral species nomenclature is the same than for nigerite. In the taaffeite group the nolanite module is modified to BeTM_4O_8 (without Sn or Ti, and lacking OH^{-1}).

In the nigerite group, in addition to the previously mentioned valid species there are known at least eight occurrences (including Roncadeira) where Zn dominates over Fe and also over (Fe + Mg+ Mn) which should therefore be identified as “zinconigerite-2N1S or -6N6S”. None of these have been formally proposed as new mineral with this name. The “nigerite” with the most prominent Zn dominance, with $\{\text{Zn}/(\text{Zn}+\text{Fe}+\text{Mn}) = 77 \text{ a } 88\%$ close to the ideal endmember composition occurs in pegmatites from Cabanas, Ponte do Lima, in Portugal and was

characterized in detail by Neiva & Champness (1997) as polysomes 6H and 24R, before the proposal of the nomenclature adopted by the IMA according to Armbruster (2002), which would now correspond to zinconigerite-2N1S or -6N6S.

Nigerite in Brazil

In this paper a first occurrence of a mineral of the nigerite group in the Borborema Pegmatite Province (BPP), Northeast Brazil, found in the Roncadeira Pegmatite, with a “Zinconigerite-6N6S” (ZN) composition, will be reported. It is the second occurrence of a nigerite group mineral in Brazil. The first one was registered by Kloosterman (1969, 1972 and 1974), found in regolith covering pegmatites, close to the Jornal village, at the Amaparí River, approximately 10 km south of the Serra do Navio Mn-deposit, in the State of Amapá, Northern Brazil. At the time of its discovery it was characterized based on two wet chemical analyses as nigerite, like other occurrences worldwide at this time. The normalization of the wt. % results of these two analyses to a sum of 66 cations would suggest a classification as “zinconigerite-2N1S” and “zinconigerite-6N6S”, respectively. Heavy mineral concentrates of the Amapá occurrences are shown in Fig. 1, together with heavy mineral concentrates from the Lixa (Seixoso) locality in Portugal, used as standard for the RRUFF data base (Downs 2006). Kloosterman (1972) informs also the finding of nigerite in two other localities in heavy mineral concentrates in the region based on optical characteristics but without chemical data.

GEOLOGICAL SETTING

Historical context

The Borborema Pegmatite Province (BPP) was known since the

begin of the twentieth century due to its production of muscovite but was studied systematically only in the mid of the century due to the increase of the exploration activity and production of beryl and Ta-ore

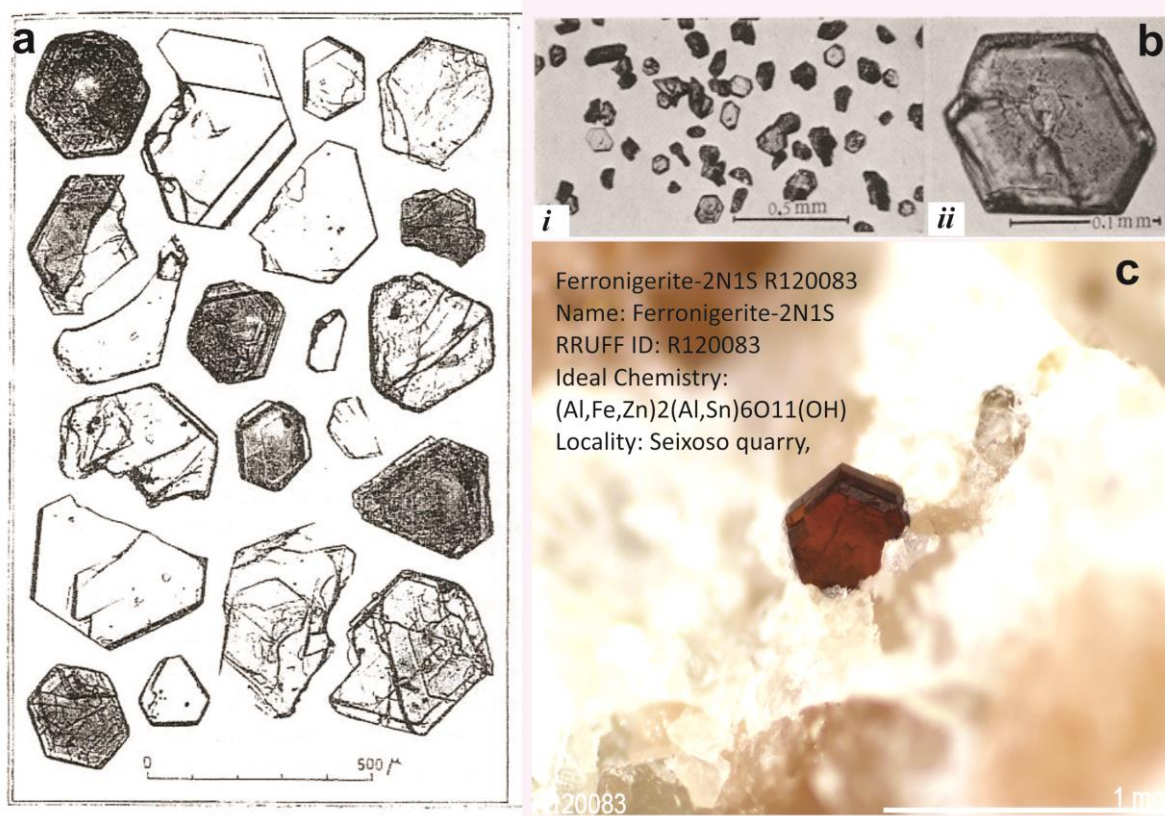


Figure 1- a) handpicked “nigerite” plates from rich heavy-mineral concentrate from the eluvial cover of a pegmatite vein from the Vila Jornal, State of Amapá according to Kloosterman (1974); b) “nigerite” concentrate rich in nearly opaque rutile (i, at the left) and individual hexagonal tabular crystal rich in inclusions (ii, at the right), from Lixa/Douro in Portugal, image by van Tassel (1975) and c) hexagonal tabular crystal (ferronigerite-2N1S) in an albite-quartz matrix, from the same locality, used as standard by RRUFF database (Downs 2006). Note that grains of the mineral are always submillimeter-sized.

concentrates, stimulated by the increasing demand of these products during the World- and Korea wars. During these years the first and still most important studies of mineralogy and geology of the pegmatites in the province were published by Rolff (1945), Johnston Jr. (1946) and co-workers. The province became famous by the discovery of some new or rare Ta-minerals, (e.g. simpsonite, Ta-aeschinite, fluor-natromicrolite) and beautiful specimens and giant crystals for collections. After a long period of declining importance due to the decrease of the Ta-ore price, more recently the BPP became important again as supplier of feldspar and caolin for the regional ceramics industry, ornamental stones (quartzites, pegmatites and pegmatitic granites), and production of the famous unique “Paraíba Tourmaline”.

Regional geologic-geotectonic setting

The BPP occupies an NNE elongated area of 150km by 70km, overlapping the eastern part of the also NNE oriented Neoproterozoic Serido Foldbelt (SFB), limited in the east by the sharp “Transpressional Picuí-João Camara Shear Zone”, with the Paleoproterozoic to Archean São José do Campestre Massif. The western limit of the SFB is a gradual (tectonically interfingered) transition of the supracrustal Neoproterozoic gneisses to the Paleoproterozoic gneisses and migmatites of the Rio Piranhas Massif. The SFB and the two massifs together compose the Northern Domain (ND) of the Borborema Tectonic Province (BTP) as defined by (Van Schmus *et al.* 2003 and Dantas *et al.* 2013). The southern limit of the ND and the BPP is marked by the transcontinental dextral shear zone known as Patos Lineament. In the north and northeast the SFB and BPP is limited by the coastal Mesozoic

sediments of the Apodi and Pernambuco-Paraíba semigraben (Fig. 2).

The SFB is composed mainly by the Neoproterozoic metasedimentary sequence of the Seridó Group, with minor metavolcanic and marble intercalations in the calcisilicatic gneisses of the basal Jucurutu Formation, quartzites, metarkoses and metaconglomerates in the intermediate Equador Formation, culminating with banded biotite-garnet-sillimanite-cordierite gneisses and schists (metaflysh) of the Seridó Formation. The Seridó-Group supracrustal rocks in the SFB overly in strong unconformity the Paleoproterozoic gneisses difficult to be distinguished from Jucurutu gneisses shown only in antiformal structures.

Several generations of granites and granitic orthogneisses are distinguished in the SFB, culminating with peraluminous pegmatitic granite S-type intrusions with still badly defined ages between 480 to 530 Ma, late or post-tectonic ages, and are the most probable source intrusions of the Be-Li-Ta-(Sn) bearing pegmatites of the BPP (Beurlen *et al.* 2014, and references therein). The pegmatites are considered to be of the REL-type according to the classification by Černý & Ercit (2005).

Local Geology

The Roncadeira Pegmatite is located approximately 500 meters south of the unpaved and partially abandoned road departing from the town of Nova Palmeira (in the State of Paraíba - PB) 4.7 km west-northwest toward the town of Parelhas (State of Rio Grande do Norte - RN). The pegmatite forms a string of several small up to 2 meters thick and a few tens of meters long, NNE oriented lenses enclosed in banded cordierite-sillimanite-garnet-biotite gneisses and schists of the Seridó

Formation. The pegmatite was known mainly for the dominance of cassiterite in the “black” ore concentrates (Rolff, 1945). According to the first description by Dottin (1961, in Roy *et al.* 1964) Roncadeira is part of a larger group of similar strings at Corredor 1 and 2, extending northward of the road (Figs. 2 and 3). The usually cassiterite-, columbite- and beryl bearing pegmatite lenses according to Dottin (1961) are parallel to sub-parallel to the foliation of the 20 to 40° NNE striking biotite schists. The subvertical to 70° dip toward ESE of the pegmatites frequently crosscuts the more gently dipping schists. The presence of

probable wodginite was first reported by Da Silva (1993 and personal communication). In addition to the cassiterite, Beurlen *et al.* (2005) distinguished ferrowodginite, ferrotitanowodginite, Ta-rich rutile (“strüverite” variety), ixiolite, tapiolite-(Fe) and gahnite as accessory ore minerals. This paragenesis was complemented by Beurlen *et al.* (2010, 2013) with the description of chrysoberyl in this pegmatite (the first one in the BPP) in association with sillimanite and presence of “zinconigerite-6N6S” a variety of the nigerite group.

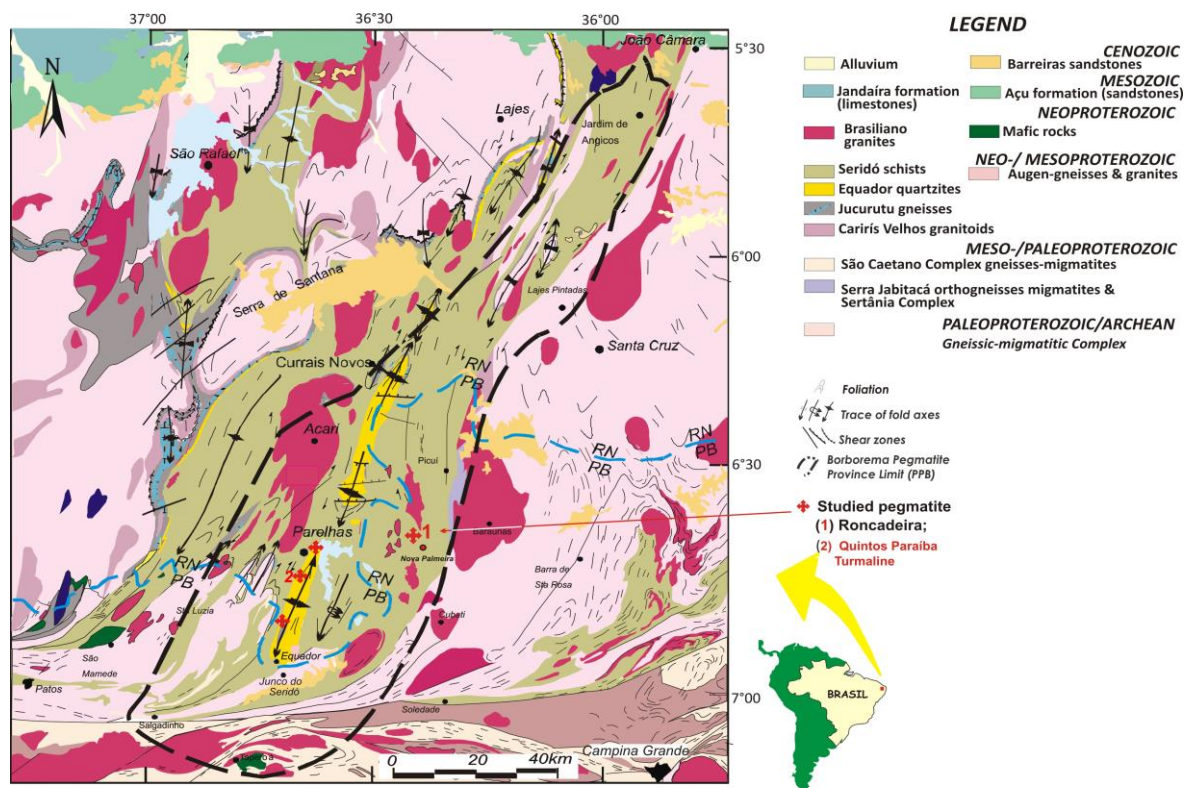


Figure 2- Simplified regional geologic map indicating the limits of the Borborema Pegmatite Province overlapping the eastern part of the Seridó Foldbelt, in the Northern Domain of the Borborema Tectonic Province according to the 1:500.000 Geologic Maps of the States Rio Grande do Norte and of Paraíba, respectively BRASIL (1998 and 2002).

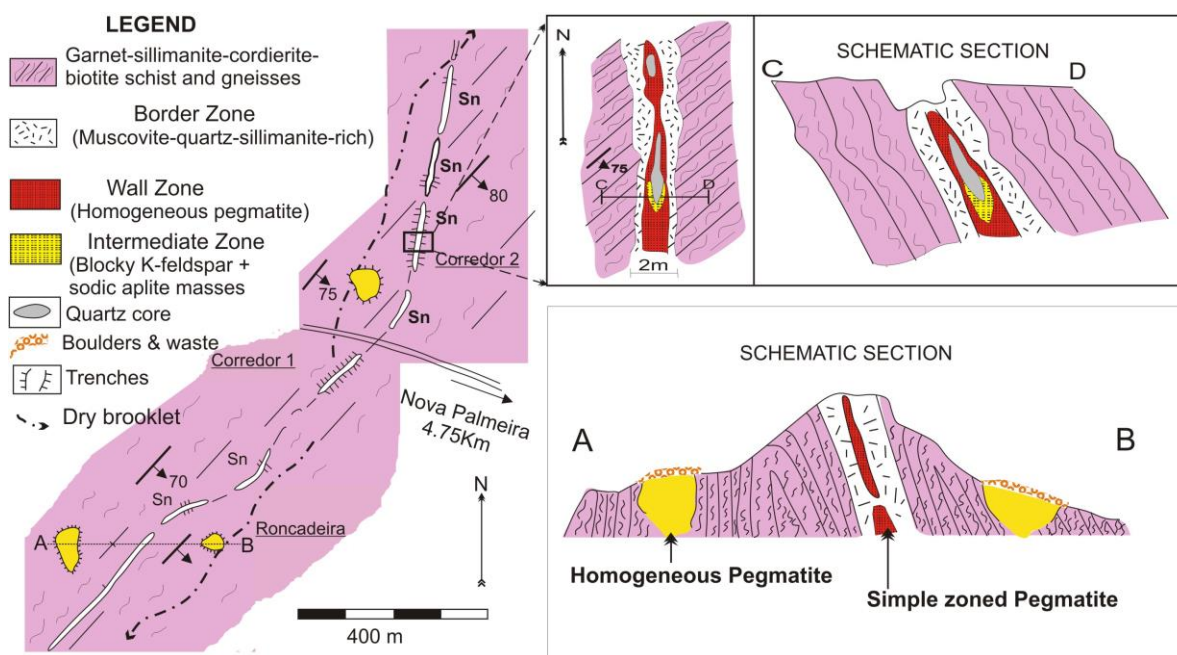


Figure 3- Local geology of the Roncadeira-Corredor pegmatite group. Sketch adapted from Dottin (1961).

Most of the pegmatite lenses of the Roncadeira-Corredor group today are almost completely covered by regolith, waste and soil. The few remaining outcrops confirm the description of the pegmatites by Dottin (1961) as almost homogeneous, poorly zoned pegmatites, with a few centimeter- to decimeter-thick muscovite-quartz±albite and locally cassiterite enriched border zone (“greisen” according to Dottin 1961).

Only one of the lenses at Corredor 2 was scavenged recently and preserves an exposition of a complete 0.0 to 2.0 meters thick cross section from the upper to the lower contact with the enclosing schists (Fig. 4). The

section allows distinguish the muscovite rich border zones, wall zone and intermittent blocky feldspar (intermediate zone) and irregular quartz pockets (discontinuous quartz core). Some small irregular masses of sacharoidal, sometimes banded albite+quartz aplites are observed both within the border zones and in the intermediate zones (replacing blocky K-feldspar). In this cross-section it is possible to see an upper pinch-out of the pegmatite lens, related with the slight shearing along the discordant contact with the schists.

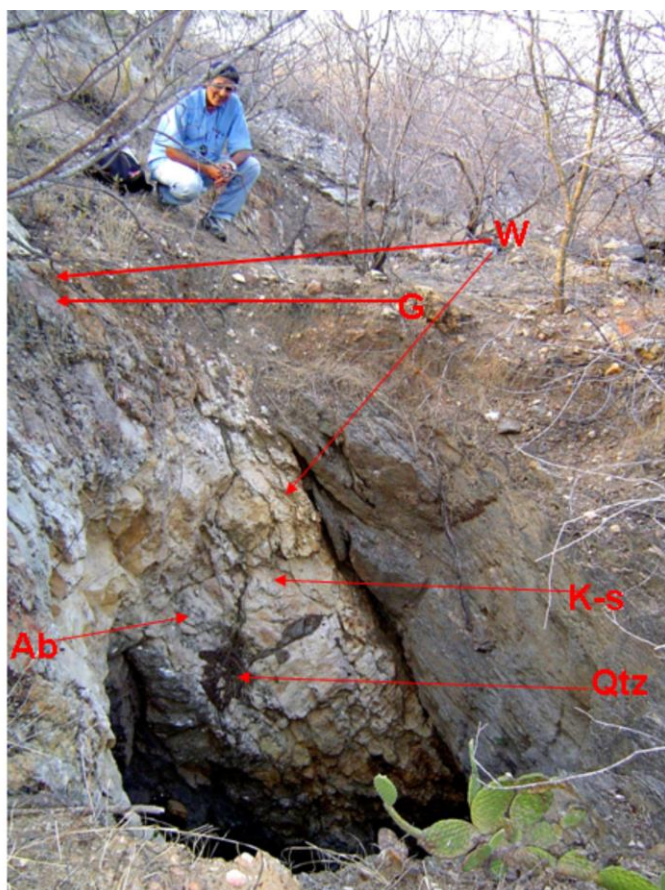


Figure 4- Trench at the Corredor 2 pegmatite lens showing the unique complete transversal section in the whole Roncadeira-Corredor pegmatite group. It is possible to distinguish the lower and upper border zones (G, “greisen” according to Dottin 1961) in contact with the schists, followed inward by the homogeneous wall-zone (W) and, only in the thickened part of the lens, by the blocky-feldspar intermediate zone (K-s) and a small irregular pocket of massive milky quartz (Qtz) possibly representing an incipient quartz core. Ab-labeled arrows indicate small masses of sodic aplite both in the intermediate and border zones. Note also the shearing related pinch-out after the upward thinning of the lens.

Petrography

The coarser grained “black ore” Sn-Nb-Ta-minerals visible with naked-eye (mainly up to 20 mm large cassiterite) seems to be concentrated in the border zones but rarely may also be seen in the wall- and blocky feldspar zone of the Roncadeira-Corredor group pegmatite lenses. In the sodic aplite masses, mainly tapiolite-, wodginite- and columbite- group- phases, gahnite, garnet and zircon are found, but occur only in submillimeter-sized grains. A boulder found in the regolith covering one of the Roncadeira pegmatite lenses represents a border zone from a sheared pinch-out portion of the lens, showing the contact with biotite-schists at both sides. In unsheared relics of sodic aplite in this boulder it was possible to identify some grains of a nigerite group mineral in addition to the usual

accessories. Fibrous sillimanite, quartz and porphyroblastic chrysoberyl found only in reddish intensively sheared parts of the sample (Figs. 5 and 6) were discussed in detail by Beurlen *et al.* (2013) and allowed to establish the temperature and pressure of its formation at approximately 600 °C and 3.5 to 5.0 kb. Preliminary EPMA (Electron Probe Micro Analyses) data in that study indicated a Zn-dominant composition of the nigerite group grains, without a detailed description.

The idiomorphic crystals of the nigerite (hexagonal shaped submillimeter-sized plates), gahnite and other Nb-Ta-minerals in the aplite suggest a crystallization in equilibrium with albite and quartz of the aplite. The relative age of crystallization of the nigerite with the main pegmatite minerals however remains not completely clear, as far as sodic aplite

masses themselves may elsewhere be formed either early and primary during the main pegmatite crystallization or late in the form of replacement bodies. In the present case, the close spatial relation with the porphyroblastic chrysoberyl along sigmoidal shear-

planes in the border zone of the pegmatite suggests a syncinematic metamorphic-metasomatic origin. Only better outcrop relations enabling a more efficient analysis of textural relations would allow to confirm or not this supposition.



Figure 5- Boulder found in the regolith covering one of the Roncadeira pegmatite lenses, showing a border zone enclosed in schists (labeled s, in the top), as also found in sheared pinch-out portions of the lenses. In unsheared relics in the sample small portions of sacharoidal, slightly banded sodic aplite (labeled a) contain some submillimetre-sized grains of a nigerite group mineral in addition to the usual accessories. The sheared portion of the sample (labeled b) contains porphyroblastic chrysoberyl crystals (green arrows) in a quartz rich aggregate (pink due to oxidized Fe-impregnation) alternating with sigmoidal microbands formed by bundles of fibrous sillimanite (pure white, highlighted by blue dotted lines).

ANALYTICAL METHODS

For the chemical characterization of the Nb-Ta-Sn-Ti-oxides in the pegmatites of the BPP a routine was established (for more details see Beurlen *et al.* 2008) beginning with X-ray diffractograms (XRD) when the individual crystal size

allows and petrography of polished sections, followed by Scanning Electron Microscopy (SEM) with backscattered electronic imaging (BSEI) and qualitative and semiquantitative analyses in selected grains, complemented by Electronprobe Microanalyses (EPMA), and in a few cases by RAMAN microspectrometry.

Formula calculations were made using Excel work-sheets for transformation of wt % results obtained with the EMPA in apfu, available in the site <http://www.open.ac.uk/earth-research/tindle/>, organized by Tindle *et al.* 2002. The same routine was used in the present study using the following Equipments: XRD data were obtained using a D 5000 Siemens X-ray diffractometer and a CuK α tube in the Department of Fundamental Chemistry of the Federal University of Pernambuco; SEM analyses for preliminary identification were obtained at the University of Campinas São Paulo using a SEM Leo 430i, Cambridge, EDS model Cat. B, using usually the following working conditions: 20 kV and 30 s acquisition time; EMPA were performed with the JEOL JXA-8500F (Hyperprobe) at the Helmholtz Centre Potsdam, German Research Centre for Geosciences, GFZ-Potsdam, Germany. Operating conditions usually were 15 kV and 20 nA, with a beam diameter of 3 mm and 30 seconds acquisition time (longer times in case of trace-elements); RAMAN spectra were recorded with a Jobin-Yvon LabRam HR800 spectrometer (grating: 1800 gr/mm), equipped with an Olympus optical microscope and a long-working-distance LMPlanFI 100x/0.80 objective. We used a 514 nm excitation of a Coherent Ar⁺ laser Model Innova 70C, a power of 300 mW (about 45 mW on sample), at a resolution $\leq 0.6 \text{ cm}^{-1}$. The spectra were collected at a constant laboratory temperature (20°C) with a Peltier-cooled CCD detector and the positions of the Raman bands were controlled and eventually corrected using the principal plasma lines in the Argon laser. The difference between the recommended and measured positions of the plasma lines in the fingerprint spectral region is not larger than 0.6 cm^{-1} .

The calculation of the nigerite group mineral formulae, with normalization to a 66 cations sum was used as suggested by Armbruster (2002), corresponding to a -6N6S polysome unit cell. The calculation was made using an adapted EXCEL work-sheet available for free use at the site <http://www.open.ac.uk/earth-research/tindle/>. In addition to the cation contents of the formulae some cation ratios were calculated for nomenclature and classification purposes. These are a) Zn/Fe and Zn/(Zn+Fe+Mg+Mn) to establish the dominance of the tetrahedral site in the spinel module of the formula, to define the prefix of the mineral-species, ferro-, magnesio- or zinco- and b) Al/(Sn+Ti) and Al/(Zn+Fe+Mg+Mn) to define the suffix, with values respectively > 7.0 and < 4.0 for the -6N6S polysomes, or if < 6.0 and > 4.0 for the -2N1S polysomes. Remembering, the Sn/ Ti values always above 1.0, typically distinguish all species of the nigerite group from högbomite group minerals.

RESULTS

Mineral-chemistry of nigerite

Data from Roncadeira

Chemical EPMA analyses of thirteen points in three grains of nigerite in the sodic aplite obtained in weight % of oxides were normalized for a total of 66 cations per formula unit (representative of 6N6S polysome unit cells) as shown in Table 1. The analysed grains are shown in the polished section of Fig 6, and, with more detail, in the BSEI in Figs. 7 and 8. The complete data, including the wt. % data of the EPMA of the nigerite and associated gahnite grains are available with the first author under request (beurlen@terra.com.br).

The standard deviation (1sd) of the average apfu of the thirteen points of nigerite are less than 11 % for Zn and Fe, 4% for Sn, 0.3 for Al, and less than

20% for minor components (Mg, Ti,) with the exception of Ta (76%). The average chemical formula is therefore representative for classification/nomenclature purpose. All data of the Zn/Fe ratio are above 1.0

and of the $\text{Zn}/(\text{Zn}+\text{Fe}+\text{Mg}+\text{Mn})$ ratio between 0.49 and 0.62 with an average of 0.56. Only three values of the grain #2 are between 0.49 and 0.50. All data therefore are Zn-dominant in the T site of the spinel module (S).

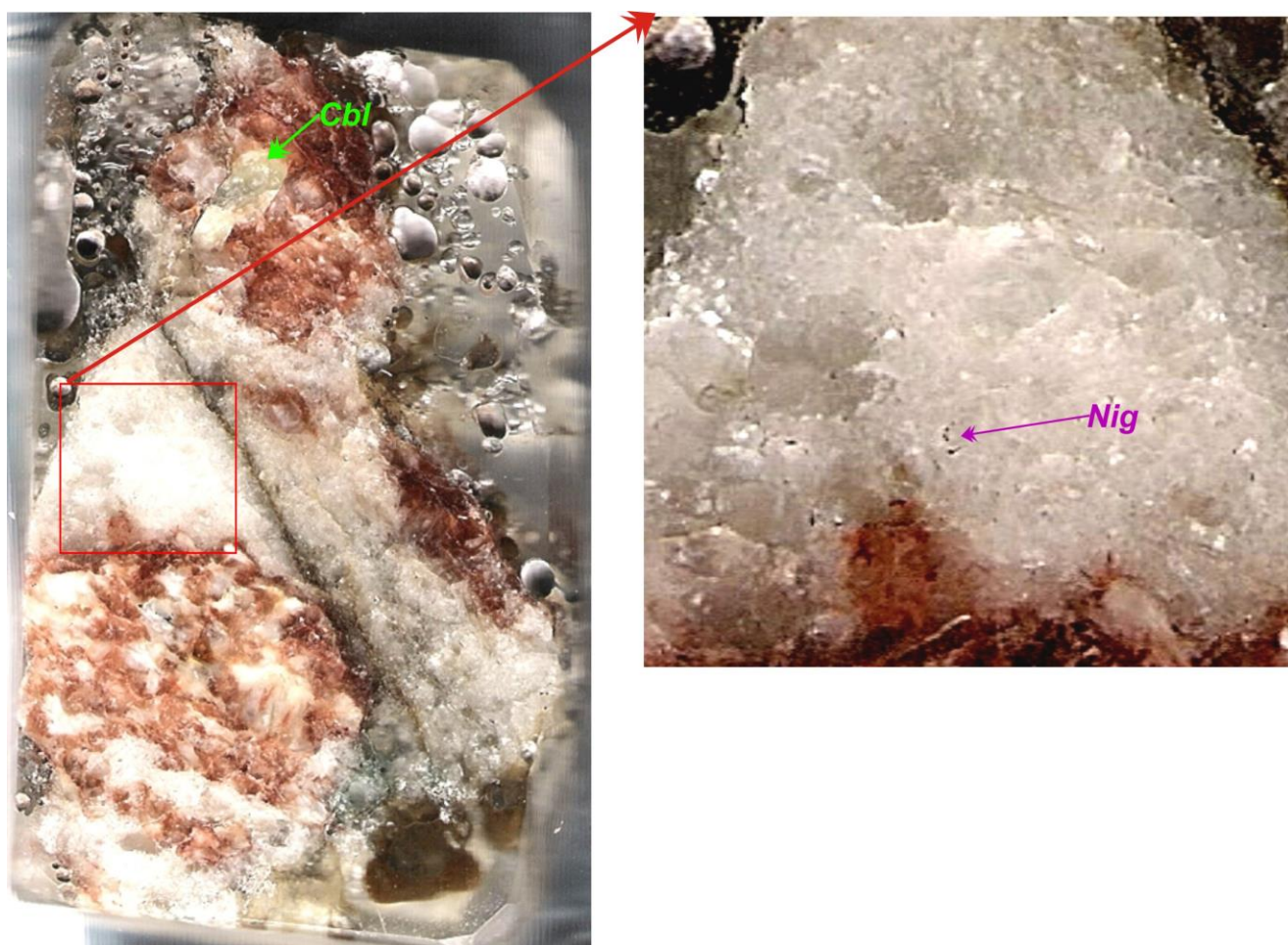


Figure 6- Tablet cut from the bolder of figure 5, used for preparation of a polished section for petrography and mineral chemistry. Chrysoberyl (Cbl, green arrow) and sillimanite occur in the quartz-rich pink colored portions. Light gray to milky sodic saccharoidal aplite relics bear several accessory Sn-Ta-Nb-Ti ore minerals, which include the nigerite-group mineral identified as a “Zinconigerite-6N6S” variety and gahnite as indicated by the purple arrow (Nig) in the magnification of the insert at the right.

Table 1 ElectronProbe Micro Analyses (EPMA) of the nigerite-group mineral from Roncadeira-sample HB-RO 24															
	sample		g2a	g2b	g2h	g2d	g2d2	g3	g3b	g3h	g3h2	g3hm	g3m2	g4	g4b
	average 1 sd		in apfu, formula normalized for a sum of 66 cations												
Si	0.66	0.146	0.80	0.72	0.58	0.59	0.91	0.52	0.54	0.48	0.50	0.69	0.74	0.83	0.83
Al	47.13	0.131	47.13	47.20	47.32	47.29	47.18	46.98	47.11	47.16	46.93	47.19	46.93	47.16	46.96
Ti	0.00	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe	4.19	0.463	4.76	4.71	4.80	4.72	4.61	3.91	3.79	3.91	3.74	3.65	3.92	3.81	3.85
Mn	0.59	0.096	0.72	0.73	0.68	0.66	0.70	0.47	0.53	0.53	0.52	0.50	0.52	0.48	0.57
Mg	0.53	0.084	0.62	0.62	0.64	0.63	0.59	0.50	0.45	0.48	0.43	0.40	0.54	0.46	0.51
Zn	6.81	0.752	5.90	5.98	5.95	6.00	5.90	7.44	7.45	7.36	7.77	7.48	7.25	7.24	7.27
Ca	0.11	0.007	0.11	0.11	0.12	0.10	0.11	0.11	0.09	0.10	0.09	0.10	0.12	0.11	0.11
Cr															
Ni	0.00	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Nb															
Sn	5.73	0.235	5.65	5.57	5.49	5.83	5.87	5.98	5.98	5.91	5.43	5.83	5.85	5.37	5.35
Ta	0.25	0.198	0.30	0.36	0.42	0.19	0.12	0.10	0.06	0.07	0.60	0.15	0.13	0.54	0.55
W															
Pb															
Total	66		66	66	66	66	66	66	66	66	66	66	66	66	66
Zn/Fe	1.63		1.24	1.27	1.24	1.27	1.28	1.91	1.97	1.88	2.08	2.05	1.85	1.90	1.89
Zn/(Fe+...	0.562		0.491	0.496	0.493	0.5	0.5	0.604	0.61	0.599	0.624	0.621	0.593	0.604	0.596
Al/(Sn+Ti)	8.225		8.34	8.48	8.621	8.109	8.031	7.858	7.879	7.977	8.65	8.1	8.022	8.785	8.781
Al/(Zn+Fe+Mn+Mg)	3.888		3.924	3.919	3.92	3.94	3.994	3.813	3.855	3.839	3.767	3.92	3.837	3.934	3.851
Polysom	6N6S		6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S	6N6S

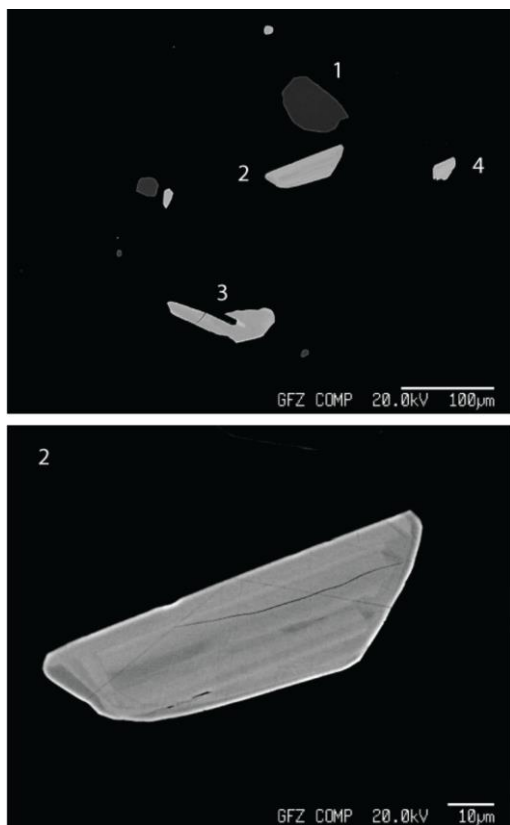


Figure 7: Backscattered electron image (BSEI) of gahnite (grain 1, dark gray) and “Zinconigerite-6N6S” (grains 2, 3 and 4 light gray, the same group indicated in Fig. 6), used for EPMA analyses and mineral chemistry determinations, in the albite-quartz matrix (black). The amplified image of grain number two shows a lamellar compositional zoning (gray tones) supposedly due to variable Ta contents.

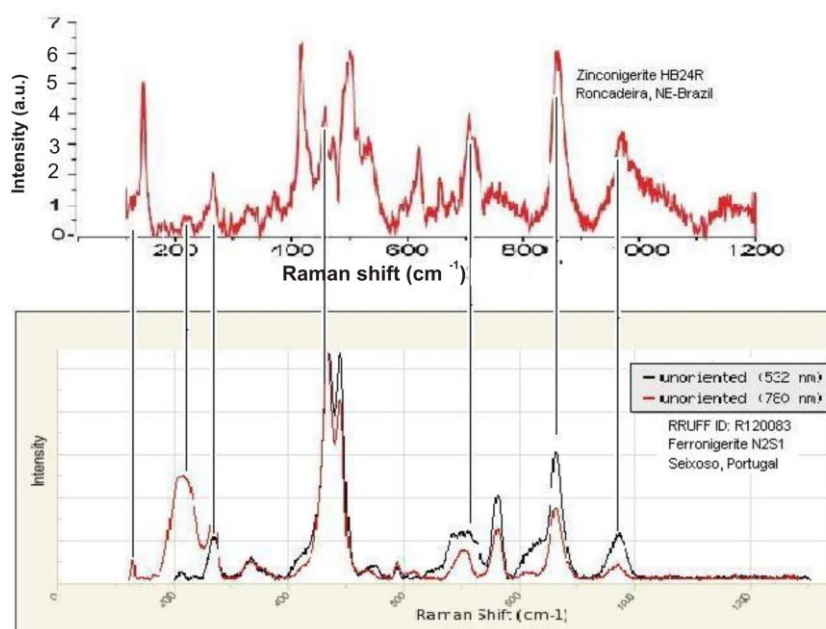


Figure 8: Raman spectrum of a grain of the “zinconigerite-6N6S” from the tablet in Fig. 6. Six peaks agree fairly well with those of the ferronigerite-2N1S of the RRUFF database (a standard of a -6N6S species is not available). Some other peaks in the sample without correspondent peaks in the standard may be due to gahnite (at 414 and 620 cm^{-1}) and albite at 500 cm^{-1}) inclusions and differences in maximal peak intensity due to mineral-chemistry variations.

In Table 2, representative data in *apfu* of all known Zn-dominant nigerite group mineral occurrences worldwide and a few Fe-dominant ones are shown together with the average formula of the Roncadeira data, for comparison. All data in Table 2 were recalculated to a 66 cation formula as suggested by Armbruster (2002) and departing from the wt. % oxide data informed by the respective authors, using the same excel work-sheet used for the Roncadeira data. This is important to allow a comparison of the data of the different occurrences because different authors could have used different normalization procedures.

The results shown in Table 2 allow the following conclusions:

- a) the recalculated formulae obtained for occurrences also recalculated by Armbruster & Feenstra (2004) are identical and therefore confirm that our calculation procedures are correct;
- b) the analyses of nigerite from the type locality referred by Jacobsen & Webb (1947), confirmed by van Tassel (1965) is a ferronigerite-2N1S, according to the nomenclature by Armbruster (2002). Nigerite from the same locality analysed by Armbruster & Feenstra (2004) is a Zn-dominant variety, that would correspond to a “zinconigerite-2N1S” , according to the new nomenclature. It is not clear if this is the consequence of a variable analytical precision or real variation of the mineral chemistry in the same locality;
- c) nigerite samples from Cabanas, Ponte do Lima, northward from the city of Porto in Portugal, studied by Neiva & Champness (1977) have the highest Zn contents reported up to now, with $Zn/(Fe+Zn+Mg+Mn)$ ratios ranging from 0.79 to 0.87 and Zn/Fe values between 3.56 and 7.65, with many data very close to an ideal zinconigerite end-member. Both polysomes 6N6S and 2N1S occur together in the same occurrence, and were identified respectively as 24R and H6 polysomes, before the new IMA nomenclature was in use;
- d) the majority (9 of 13) of pegmatite hosted or related nigerite occurrences are Zn-dominant, or bear both Zn- and – and Fe-dominant nigerite species. None of these are Mg-dominant with Mg values, which always amount less than 10% of the T site in the spinel module. The majority of the cases (8 of 13, including the type locality) have Zn/Fe ratios higher than 1.0 and the classification as ferronigerite polysomes would be inadequate;
- e) in several occurrences Zn- and Fe-dominant species occur together; this is the case in the type locality, in Rosedal (Finland), and Separation Rapids (Canada); the same is valid for coexistence of 6N6S and 2N1S polysomes, as in the type locality, Cabanas (Portugal), Amapá (Brazil), and Separation Rapids (Canada)

Table 2 Comparison of stoichiometric formulae of nigerite-group minerals in the main pegmatite-related occurrences worldwide

Neiva & Champness 1997					Armbruster & Feenstra 2004		Novk et al	2009	van Tassel 1965			Kloosterman, 1974		Burke et al77	Čech 1978	Tindle & Breaks 1998				Beurlen	
Cabanas, Portugal, "zinco nigerite"					Omaruru Namibia	Egbe Nigeria	Portugal			Portugal Lixa	Nigeria	Siberia	Brazil Amapá		Finland	Czech Republic	Separation Rapids Canada				Roncador eira-Brazil
1l	2l	3l	2h	3h			17	13					Eluv	Peg	aplito	granito	peg1	peg9	peg1b	peg10	averag
Recalculated from wt.% wet chemical and Electron Probe Microanalyses (EPMA) to a 66 cation sum normalized formula																					
Si	0,35	0,62	0,74	0,72	0,88	0,06	0,04	0,00	0,00	0,65	0,36	2,99	0,15	3,30	0,21	0,37	0,00	0,00	0,00	0,00	0,66
Al	46,66	46,77	46,77	47,73	47,82	46,74	46,50	46,44	46,39	47,16	44,91	48,52	46,44	44,09	46,54	46,24	47,91	47,23	47,05	46,70	47,13
Ti	0,14	0,30	0,37	0,60	0,80	1,22	0,83	2,34	0,58	1,81	0,10	0,27	0,00	0,00	0,08	0,59	0,05	1,36	0,04	0,23	0,00
Fe	1,47	1,63	1,67	1,64	1,90	5,94	5,95	7,26	7,36	6,63	9,10	3,56	4,83	5,83	5,95	6,64	6,99	7,39	5,66	6,84	4,19
Mn	0,06	0,11	0,11	0,13	0,08	0,17	0,10	0,52	0,57	0,06	0,06	0,07	0,00	0,00	0,21	0,49	0,39	0,25	0,28	0,34	0,59
Mg	0,00	0,00	0,00	0,00	0,00	0,75	0,80	1,39	1,29	0,11	1,43	0,53	0,00	0,00	0,63	0,00	0,00	0,13	0,09	0,09	0,53
Zn	11,25	10,81	10,69	7,26	6,79	6,05	6,36	4,41	3,94	2,85	2,49	4,62	5,32	6,50	6,25	6,22	1,82	2,03	6,67	5,71	6,81
Ca	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	nd	0,09	0,00	0,00	0,00	0,00	0,11
Cr	0,00	0,00	0,00	0,00	0,00	0,00	0,01	0,00	0,00	0,00	0,00	0,00	0,00	0,00	nd	0,00	0,00	0,00	0,00	0,00	0,00
Ni	0,00	0,00	0,00	0,00	0,00	0,01	0,01	0,01	0,01	0,00	0,00	0,00	0,00	0,00	nd	0,00	0,00	0,00	0,00	0,00	0,00
Nb	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,07	0,00	0,05	0,00	0,11	0,12	0,05	0,12	0,00
Sn	6,07	5,76	5,64	7,92	7,73	5,06	5,41	3,61	5,84	6,73	7,56	5,44	8,78	6,29	5,99	5,28	8,56	7,27	6,10	5,77	5,73
Ta	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,02	0,02	0,00	0,00	0,00	0,41	0,00	nd	0,07	0,08	0,16	0,04	0,17	0,25
W	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	nd	0,00	0,09	0,04	0,04	0,04	0,00
Pb	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	nd	0,00	0,00	0,00	0,00	0,00	0,00
Σ cations	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	0	66	66	66	66
Zn/(Σ T ^{sp})	0,88	0,86	0,86	0,80	0,77	0,47	0,48	0,32	0,30	0,30	0,19	0,53	0,52	0,53	0,48	0,47	0,20	0,21	0,53	0,44	0,56
Al/(Sn+Ti)	7,51	7,71	7,78	5,61	5,60	7,44	7,45	7,81	7,22	5,53	5,87	8,50	5,29	7,01	7,67	7,88	5,56	5,47	7,67	7,79	8,22
Polysome	6N 6S	6N 6S	6N 6S	2N 1S	2N 1S	6N 6S	6N 6S	6N 6S	6N 6S	2N 1S	2N 1S	2N 6S	2N 1S	6N 6S	6N 6S	6N 6S	2N 1S	2N 1S	6N 6S	6N 6S	6N 6S
Al / Σ T ^{sp})	3,65	3,73	3,75	5,28	5,45	3,62	3,52	3,42	3,52	4,89	3,43	5,52	4,58	3,58	3,57	3,46	5,21	4,82	3,71	3,60	3,89
Zn/Fe	7,65	6,65	6,42	4,42	3,58	1,02	1,07	0,61	0,54	0,43	0,27	1,30	1,10	1,12	1,05	0,94	0,26	0,27	1,18	0,83	1,62

Several cases of nigerite-group mineral occurrences in geological context other than hosted in or directly related with pegmatites may be distinguished:

- a) at the contact of granites with Devonian marbles in China like magnesian nigerite-6N6S (Chen *et al.* 1989, e Yang *et al.* 2013a), or Li-rich ferronigerite-2N1S (Yang *et al.* 2013 b);
- b) in sulphide mineralized high grade metamorphic rocks at Falun in Sweden (Shumacher *et al.* 1987), and at Geco, Manitouwadge, Ontario Canadá (Essene *et al.* 1982, Spry. 1982);
- c) as metamorphic mineral in cordierite-staurolite-gedrite-hercinite-gneisses at Benson-Manitouwadge, Ontario (Petersen *et al.* 1989). The same authors also discuss the conditions for the formation of högbomite-group mineral occurrences usually considered to be of metamorphic origin;
- d) taaffeite-group minerals, also belonging to the högbomite supergroup, are still less common than nigerite and usually considered to be the result of metamorphism in pegmatites. Sometimes they occur forming very rare and expensive gemstones, mostly as product of exomorphic contact reactions related to pegmatite or granite interaction with skarns or marbles (Armbruster 2002, Schmetzer *et al.* 2005, 2007, and references therein).

A more detailed discussion of the högbomite- and taaffeite- group mineral occurrences and not pegmatite related nigerite group mineral occurrences is beyond the scope of this study.

Comparison of geological and textural features of pegmatite related nigerite (NG) occurrences

A summarized description of the main geological, mineralogical and textural features of all pegmatite-hosted or -related nigerite-group (NG) mineral occurrences is listed in Table 3. Most common features are the presence of Zn-dominant nigerite (8 of 10 occurrences, only two of them bear

Fe-dominant species alone); occurrence of the NG in the greisen-like (rich in albite, quartz and muscovite) border-zone, association with sillimanite or andalusite (7 in 10); association with cassiterite (9 of 10); association with CGM (8) association with gahnite (06 of 10), association with chrysoberyl (5).

The preferential occurrence of the NG in the border zone and association with aluminosilicates is suggestive of a late- or post-pegmatitic, syncinematic-metamorphic crystallization. The fact that in table 3 there is no positive reference of a paragenetic relation with aluminosilicates in some occurrences may be due simply because most publications are of purely mineralogic-crystallographic nature, and detailed textural information are omitted or even lacking (e.g. many samples are components of heavy mineral concentrates). Only in a few occurrences, NG are clearly described as idiomorphic inclusions in typically primary pegmatite minerals. Therefore, it is probable that there are two genetic types of pegmatite related NG occurrences: a primary one of crystals formed early and still in equilibrium with the pegmatite melt (inclusions in primary pegmatite minerals) and, more commonly, as late-stage, syncinematic-metamorphic metassomatic occurrences, in greisen-like border zones or late-stage metassomatic-hydrothermal replacement bodies within pegmatites.

In the case of Roncadeira Beurlen *et al.* (2013), considering the presence of NG and gahnite occurring together in the border zone, less than 1cm apart from porphyroblastic chrysoberyl + sillimanite + quartz in sigmoidal shear zones, a late- or post-pegmatitic syn-cinematic-metamorpho-metassomatic origin at 600°C and 3.5 to 5.0 kb is supposed.

Table 3: Comparative summary of geological, mineralogical, textural features of pegmatite related nigerite-group mineral occurrences.										
Name/locality	Cabanas Portugal	Barroso Portugal	Lixa Portugal	Amapá-Brazil	Eggbe Nigeria	Namíbia	Finnland	Sep. Rapids Canadá	BohemiaC Czech Rep	Roncadeira NE-Brazil
¹ Reference: first uthor	Neiva	Novak &	v. Tassel	Kloosterman	Jacobsen &	Knorring	Burke &	Tindle &	Čech et.	Beurlen et al.
veins close to ²peg's,		close to peg's							close to peg's	
Host rock	in peg	quartz vein	in peg's (+ aplites)	in peg's	³ "greisen" + in peg's	peg	peg's (+ aplites)	in pegs	peg facies in granites	peg (border) + sheared aplite
Wall rock	schists	schists	-	schists	schists & gneisses	-	gabbro	meta-mafic	granite	schists & gneisses
Exocontact				³ "greisen" = Qtz-sill rock	"greisen" = Qtz-sill rock	Qtz-sill rock				"greisen"
Paragenesis ⁴ dom cat in nigerite* gahnite sillimanite andalusite cordierite chrysoberyl rutile cassiterite columbite-group CGM apatite Turmaline ⁵ staurolite(Zn) ⁶ "Squi"	Zn-yes yes yes yes yes yes yes yes yes yes yes	Fe- yes yes yes yes yes yes yes yes yes	Fe-yes yes yes yes yes yes	Zn-yes ± yes yes yes yes yes yes yes yes	Fe- + Zn-yes yes yes yes yes yes yes yes yes	Zn-yes yes yes yes yes yes yes staurolite(Zn)	Fe- + Zn-yes yes yes yes yes staurolite(Zn)	Fe- + Zn-yes yes yes yes yes "Squi"	Zn-yes yes- yes yes yes yes yes	Zn-yes Yes sim sim sim sim sim
replaces & overgrows	gahnite									
Explanations: ¹ only the first author names are mentioned, complete references in the text; ² peg abbreviation of pegmatite; ³ "greisen" used by authors as equivalent of border zone or "quartz+sillimanite rock" in reaction with wall rock; ⁴ the dominant cation in the T-site of the spinel module (S); ⁵ staurolite with anomalous high traces of Zn; ⁶ "squi" = pseudomorph aggregate of quartz and spodumene formed by break down of petalite.										

CONCLUSIONS

“Zinconigerite-(6N6S)” (ZN) was found as rare accessory submillimeter sized grains in the border-zone of the Roncadeira Pegmatite in the Nova Palmeira County, State of Paraíba. It is the first occurrence of a mineral of the nigerite group in the Borborema Pegmatite Province, in Northeast Brazil, and second occurrence in Brazil. The ZN at Roncadeira is associated with gahnite, other Ta-Nb-Sn-Ti oxides, chrysoberyl and sillimanite in small masses of saccharoidal, slightly banded sodic and sheared aplite. The texture relations of the porphyroblastic chrysoberyl-fibrolite pair suggest the syncinematic-metamorphic-metasomatic crystallization at approximately 600° and 3.5 to 5.0 kbar. The mineral association found at Roncadeira is similar to other seven of nine occurrences of the “Zinconigerite-6N6S” variety of nigerite group minerals worldwide. The high P/T crystallization conditions suggest the possibility of the existence of another, earlier generation of rare element pegmatites in the province because those Be-Li-Ta bearing pegmatites known up to now are supposedly formed during a late- to postectonic stage at lower regional temperature conditions.

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