



The Serra do Caparaó Complex, Mantiqueira Province, Brazil, revisited: metamorphic age constraints by U-Pb and Lu-Hf method in zircon by LA-ICP-MS

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Abstract

The U-Pb method was used in order to understand the high-grade metamorphic rocks in the Ribeira Belt (SE Brazil) and Lu-Hf isotopes were applied to investigate the magmatic protolith sources and the tectonic setting of Serra do Caparaó Complex rocks. The results indicate the existence of two thermal events: the first related to a Paleoproterozoic magmatic event and the second one linked to high-grade metamorphism related to the Brasiliano Orogeny. The U-Pb age interval from 2209 to 2060 Ma obtained in zircon grains from the granulitic orthogneiss with $T_{DM_{crustal}}$ ages ranging from 2.13 to 3.87 Ga may be interpreted as mantle-derived source with important older crust participation in the magma formation; juvenile arc-related rocks were generated at this time as indicated by the positive ϵ_{HF} values. The second group of U-Pb ages ranging from 633 to 584 Ma is interpreted as a metamorphic event, resulting from the collisional process related to the Gondwana assembly.

Article Information

Publication type: Research Papers

Received 1 May 2021

Accepted 27 December 2021

Online pub. 7 January 2022

Editor: Evandro Klein

Keywords:

U-Pb isotopes

Lu-Hf isotopes

LA-ICP-MS

Zircon

Ribeira Belt

Gondwana assembly

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1. Introduction

Geochronological studies have become, in the last decades, an indispensable analytical tool for geosciences studies. U-Pb and Lu-Hf isotope analytical techniques in zircon have been gradually improved, thus enabling the investigation of several geological processes that occur in crustal dynamics (Santosh et al. 2009, Andersen et al. 2002). The study area of this work is located in Serra do Caparaó region (Figure 1), at southern of the Araçuaí Orogen and included in the northern sector of Mantiqueira Province (Pedrosa-Soares et al. 2001). The lithological associations of Serra do Caparaó region were correlated in the literature to the Juiz de Fora Complex and should represent the basement rocks of the Ribeira-Araçuaí belts, of Rhyacian age (≈ 2.19 Ga), according to Campos Neto and Figueiredo (1990).

The evolution of the basement of the Ribeira-Araçuaí belt occurred as a result from the agglutination of Archaean crustal

blocks during the Paleoproterozoic orogenic processes, which were evidenced in the period from 2.2 to 2.0 Ga. These Archaean-Paleoproterozoic blocks represent, according to Heilbron et al. (2004), remnants of a magmatic arc developed in the Archaean paleocontinent margin, and of one or more additional magmatic arcs, which were dismembered and reworked in the Neoproterozoic era by the Brasiliano Orogeny.

Although the nature of the rocks from Serra do Caparaó region is still a matter of great debate for the scientific community, although these are treated by some authors as a unit of the basement of the orogen (Seidensticker and Wiedmann 1992), other authors believe that it is part of the Juiz de Fora Complex (Campos Neto and Figueiredo 1990). Compositionally, there is an association of charnockite, diorite, and gabbro defined by Horn (2006) as Carapaó Suite, with Paleoproterozoic (2.2 Ga) U/Pb ages (Silva et al. 2002). The migmatitic unit of the Caparaó Suite shows various intensities of partial melt. The



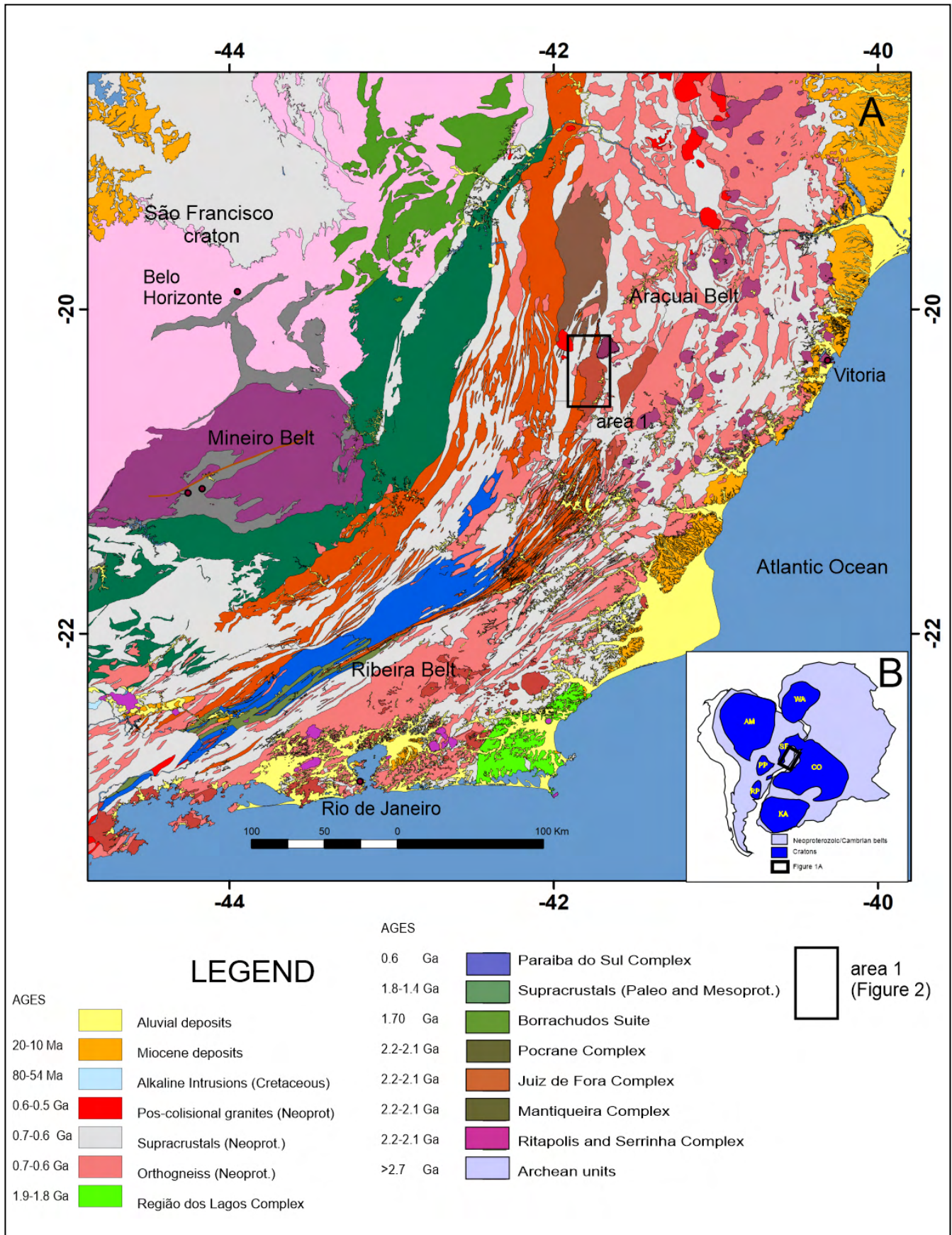


FIGURE 1. (A) Regional geologic map of the studied area in the context of Ribeira and Araçuaí belts (adapted from Machado et al. 2021). (B) Paleogeographic reconstruction of West Gondwana. The study area is signed (rectangle).

main migmatitic structures are schlieren, ptigmatic, agmatic and complex folded rocks (Horn 2006). Two events affect the rocks of this unit, the first Paleoproterozoic in age (ca. 2.2-2.1 Ga), which is responsible for the formation of diagnostic paragenesis of granulite facies (orthopyroxene + plagioclase \pm clinopyroxene \pm hornblende) as reported by Noce et al. (2007) to high-grade metamorphism. This metamorphic event was broadly coeval with the magmatic crystallization. The second event is related to the Brasiliano Orogeny, whose collisional episodes led to the amalgamation of different tectono-stratigraphic terranes that progressively collided against the southeastern margin of São Francisco Craton (SFC) between the Early Ediacaran and the Cambrian (Heilbron et al. 2000, 2004, 2008, 2020; Trouw et al., 2000; Schmitt et al. 2004, 2008, 2016; Freitas et al. 2021), where the regional foliation generated is evidenced by the crystallization of hornblende, biotite and garnet and locally orthopyroxene and clinopyroxene.

The present work aims to investigate the crystallization and metamorphic ages of granulitic rocks of Caparaó Complex, providing information about the crustal evolution of this terrane in the internal tectonic domain of the Araçuaí Orogen. Thus, the aim of the study herein is to contribute to the understanding and characterization of the Paleoproterozoic and Neoproterozoic evolution of the Ribeira-Araçuaí belt and the border of the São Francisco Craton, by constraining the U-Pb zircon ages in the Caparaó Complex to define the crystallization ages of the granulitic rocks photoliths. In addition, Lu-Hf analysis were performed to provide the sources of these granulitic lithotypes.

2. Regional Geology

The units observed in the study are included in the Araçuaí Belt as defined by Almeida (1977, 1981) and comprise the eastern portion of the São Francisco Craton (Silva et al. 2002, 2005; Noce et al. 2007; Pedrosa-Soares 1992, 1999, Amorim et al. 2021). Its origin is related to the Brasiliano Cycle, which comprised a series of collisional events, from the Neoproterozoic era lasting to the Paleozoic era, when the passive margin of the São Francisco paleocontinent was amalgamated with the active margin of the Congo paleocontinent (Figure 1B) (Seindensticker and Wiedeman, 1993; Vieira 1997; Horn 2006; Novo et al. 2011; Vieira and Menezes 2014; Pinto and Silva 2014; Vieira and Menezes 2014).

In the study region, the Araçuaí Belt has its southern extension represented by rocks from Juiz de Fora/Paraíba do Sul Domain (Pedrosa-Soares et al. 2007; Novo et al. 2011). This Domain occupies an extensive northeast-oriented belt throughout the central-north and western portion of Rio de Janeiro state and SE of Minas Gerais state, subparallel to Rio Paraíba do Sul shear Zone. It consists of kinzigite gneisses, schists, quartzites and marbles from Paraíba do Sul Complex (Angeli 1978), metamorphosed into amphibolite and granulite facies, tectonically intercalated in Paleoproterozoic rocks represented by tonalitic orthogranulites and orthogneisses from Juiz de Fora Complex, and granitic to granodiorite orthogneisses from Quirino Suite.

In the Araçuaí Orogen, several geotectonic components are found, such as: passive margin deposits, ophiolitic splinters, magmatic arc and associated basins, syn-collisional granites, post-collisional granitoids, among others, which together mark the evolution of an accretionary orogen to the collisional orogen stage (Correia Neves et al. 1987; Pedrosa-Soares et al.

2001, 2007). Therefore, the Araçuaí Belt is divided, according to Pedrosa-Soares et al. (2001) in External (West) and Internal (East) domains. The external domain comprises the folding-thrusted belt, of low metamorphic degree, and the internal domain is characterized by the granitoids related to different evolutionary stages, reaching a high metamorphic degree.

The metamorphism associated with the Brasiliano collisional event reworked older rocks with Paleoproterozoic to Archean ages, notably rocks from the Mantiqueira and Juiz de Fora complexes, and from the orthogneisses of the Quirino Unit. These units constitute inliers of the basement of the province, tectonically intercalated with the metasedimentary rocks of the Andrelândia Group.

In the Espera Feliz region, three dominant units can be identified with relative lithological homogeneity: the basement, the Neoproterozoic metasedimentary cover and the Neoproterozoic-Cambrian granitoids (Wiedemann 1993; Noce et al. 2007; De Campos et al. 2004). The study area of this work is focused on the internal domain, with dominant tectonic trends in the NNE directions with a westward vergence. In this domain, the Neoproterozoic and Cambrian granitoid suites are hosted by basement rocks (Pedrosa-Soares et al. 2001, 2007).

3. Local Geology

The Caparaó Complex (Figure 2) comprises a unit formed by granulitic orthogneiss from felsic to intermediate composition which is the predominant lithology in the work area but variations may also occur with clinopyroxene and amphibole and units of mafic composition (Campos Neto and Figueiredo 1990; Pedrosa-Soares et al. 2001, 2007). Metasedimentary rocks are represented by the Andrelândia unit, which is composed of paragneisses and quartzites, and interpreted as deposited in a platform basin (Paciullo et al. 2000; Ribeiro et al. 2012). Isotropic granites are regionally described as post-orogenic granites and may represent the collapse period of the orogenic process. The post-collisional stage (ca. 530-480 Ma) is formed by G4 and G5 supersuites, which consist from calc-alkaline to alkaline, A and I-type granitoids, without deformation features (Wiedemann et al. 1987; 2002; Pedrosa-Soares et al. 2011; De Campos 2014; De Campos et al. 2016).

The granulitic orthogneisses were divided in two groups according to the mineralogical composition; the first group presents mainly granulitic gneiss with felsic to intermediate composition (charno-enderbitic), occurs with gray to dark gray coloration or grayish when it presents certain degree of weathering. The compositional banding continuity is observed in large outcrops, occurring folded locally with axis filled with melt (Figure 3 A and B). Garnet may be observed as large crystals (Figure 3C) or in banded gneisses with thickness from millimeters to centimeters. Large enclaves are locally observed. In thin sections, banded gneisses are marked by mineral segregation in felsic bands, with quartz, plagioclase, and rare K-feldspar; and in mafic bands, with pyroxene (Figure 4A and B), amphibole, biotite, and garnets. Zircon and oxides are accessory minerals.

A second group of granulitic orthogneiss is comprised of felsic rocks presenting quartz and plagioclase (Figure 3 D) that occur with granoblastic texture and have fine to medium grain size, while K-feldspar occurs as porphyroclasts up to two centimeters. Garnet grains occur mainly in felsic bands as crystals of about 1 centimeter but they may occur agglomerated. Amphibole crystals occur in the felsic bands (Figure 4C and

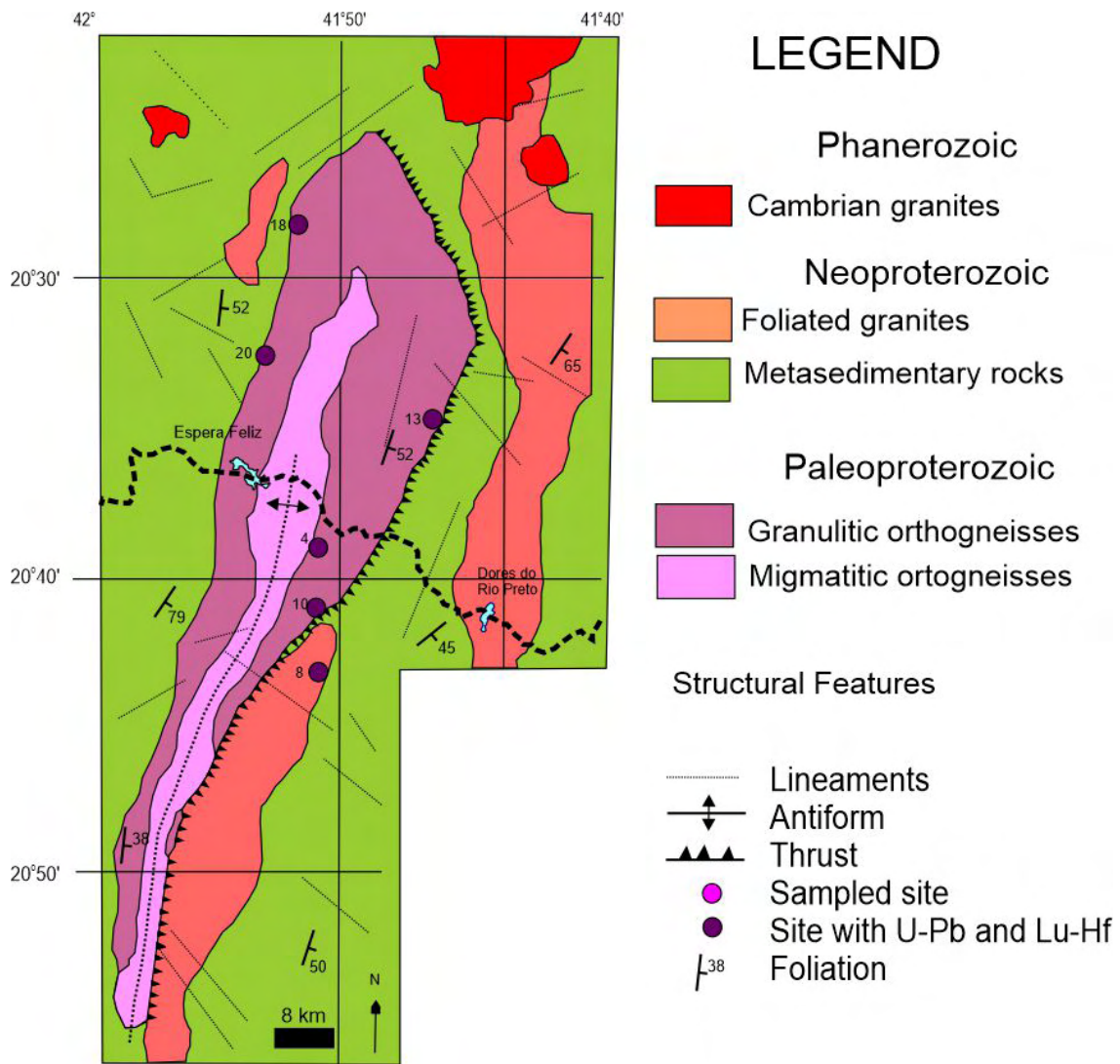


FIGURE 2. Geological map of Serra do Caparaó study area. The sampled points are shown on the map.

D), as porphyroclasts and in the mafic bands the pyroxene occurs with fine to medium-sized with nematoblastic texture. The mafic orthopyroxene gneiss appears mainly in the higher elevations of the Caparaó Complex, as it enclaves in the middle of the orthogneiss migmatized unit, presents dark color and mafic composition, with or without millimetric banding. Centimetric to metric mafic enclaves are often observed (Figure 3 E), locally stretched in the banding direction (Figure 3 F). In contact with other units in the area, the gneissic rocks of the Caparaó Complex develop a complex folded features characterized by open and tight folds (Figure 5 A, B, C and D).

4. Materials and Methods

Seven samples were taken to collect representative rocks of the units observed in Serra do Caparaó region (Fig. 2). In this way, the samples were included in two groups (Table 1): the first is composed of charno-enderbitic gneisses (samples CPR03, CPR04, CPR08 and CPR10) and the second group is composed of felsic orthogneiss (samples CPR13, CPR18 and CPR 20).

4.1. Sample preparation and Scanning Electronic Microscopy (SEM)

Initially all the collected samples were washed and dried to avoid any contamination for geochronological studies. The crushing was performed on a jaw crusher that breaks the rock to sizes of approximately 1 centimeter. This was followed by disc-mill to obtain fined-grained material and then by gravimetric separation is carried out with a ruffled table. This procedure aimed to separate the heavy minerals. Finally, the concentrated heavy minerals were taken to separation in dense liquid and magnetic separation. This procedure was performed to find larger amounts of zircon grains with different magnetic susceptibilities, followed by the epoxy preparation in which the zircon grains were fixed to an epoxy resin. After drying the resin, the samples were sent to the polishing process and then to the Scanning Electron Microscope (SEM) QUANTA 250 using backscattered and cathodoluminescence detectors for imaging of the zircon grains and observation of internal mineral structure and evidence of inherited core, areas of reabsorption, magmatic zoning to guide the subsequent location of the isotopic analysis.

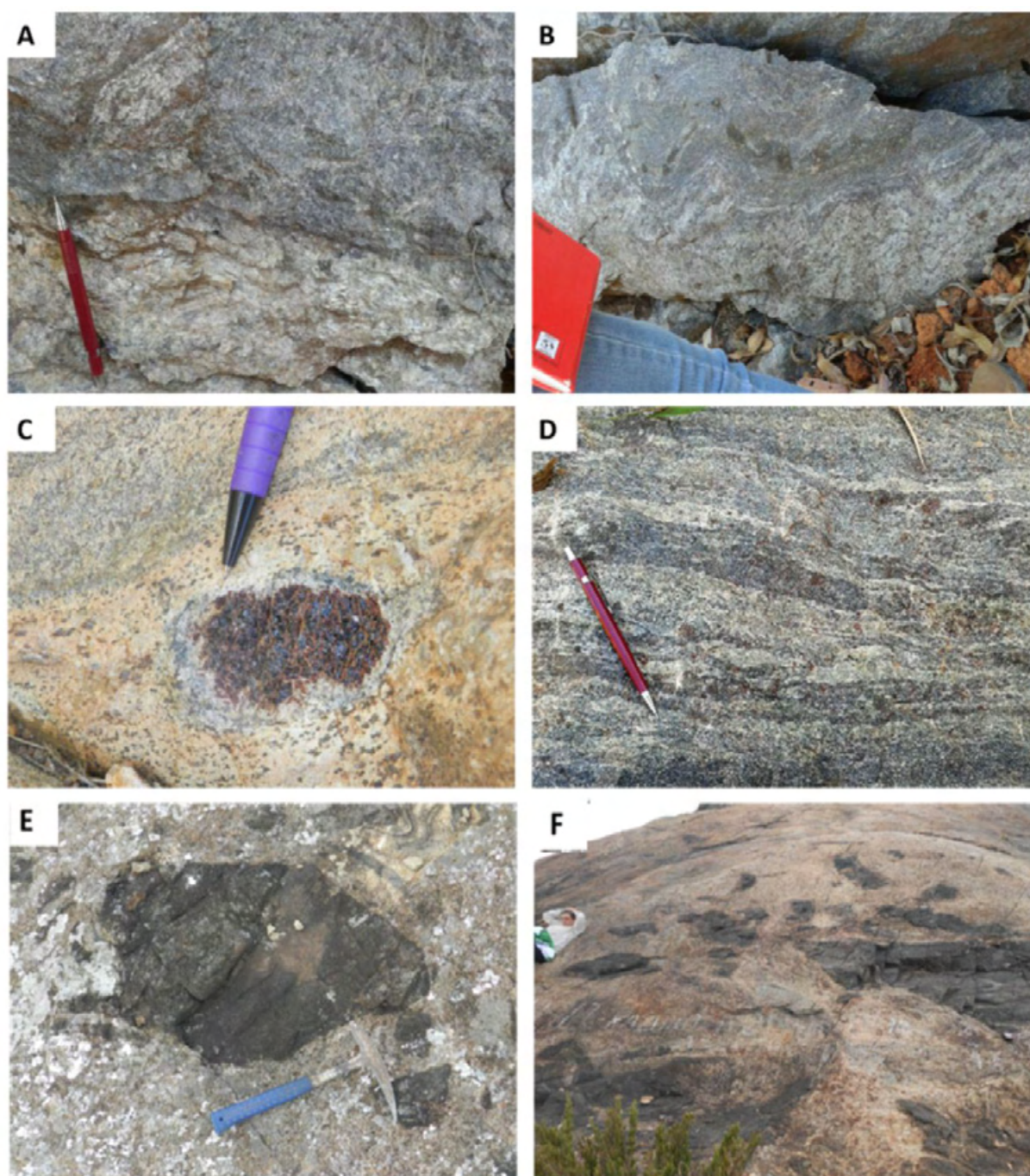


FIGURE 3. Outcrop images of orthogneisses and migmatitic rocks of the Caparaó Complex. (A) and (B) Local melt; fold with axes filled by melt. (C) Garnet; (D) Layers of garnet; (E) and (F) Enclaves observed in banded gneiss.

4.2 U-Pb dating

The spectrometer used to perform the isotopic quantification of the present work was a Neptune plus (Thermo Finnigan) in the MultiLab laboratory, of Rio de Janeiro State University. It is a high-resolution Multi-Collector Induced Coupled Plasma Mass Spectrometer (MC-ICP-MS) for isotopic measurements, with a special configuration to simultaneously detect a wide range of element nuclides using 9 Faraday collectors and 6 ion counters which can be combined in an appropriate configuration for the isotopes of interest for the U-Pb method (Jackson et al. 2004; Geraldés 2010). During the analysis, the collector's geometry (with 2 Faradays and 5 ion count)

followed the specifications presented in Table 2. The U-Th-Pb isotope analysis includes ^{204}Pb , ^{206}Pb , ^{207}Pb , ^{208}Pb , ^{232}Th , and ^{238}U . Mercury represents a common contaminant in He and Ar gas and ^{204}Hg interferes with ^{204}Pb mass counts. Mass ^{202}Hg , mass $^{204}({}^{204}\text{Pb} + {}^{204}\text{Hg})$ as well as ^{206}Pb , ^{207}Pb and ^{208}Pb were detected by the ion counters while ^{238}U and ^{232}Th were measured with a Faraday cup. Isotopic data was obtained using the static mode through 40 cycles of 1.054 seconds acquisition time with a gas inlet flow (Ar) of 15 L / min, auxiliary flow (Ar) 0.8 L / min, in MC-ICP-MS (Table 3).

The acquisition process consisted of a sequence (Figure 6), which began with the analysis of one blank, which is the data measurement performed in the passage of only Ar and

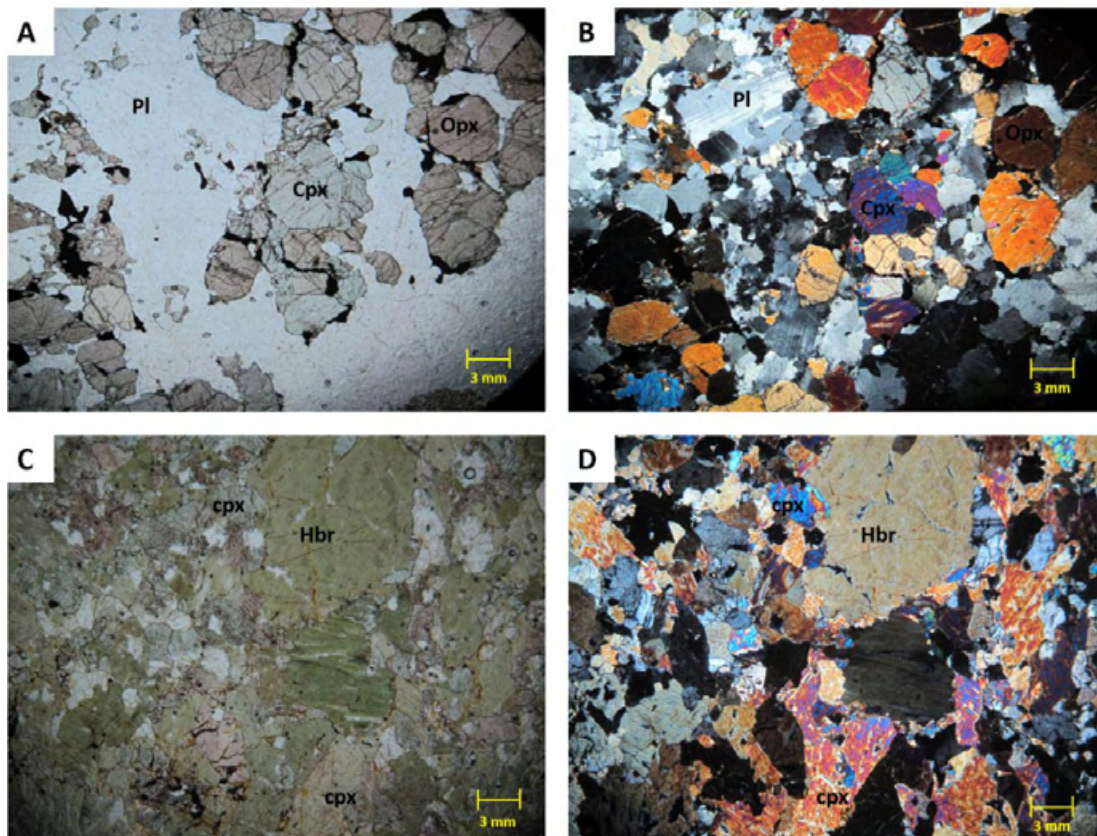


FIGURE 4. Photomicrographs of some points where the clinopyroxene (Cpx) - orthopyroxene (Opx) gneiss variation predominates in the granulite orthogneiss unit. (A) and (B) granoblastic arrangements of Cpx, Opx, plagioclase (Caparaó region sample CPR 04). (C) and (D) Clinopyroxene associated with hornblende. Legend Minerals: Cpx- clinopyroxene; Opx- orthopyroxene; Hbr - hornblende, Pl - plagioclase.

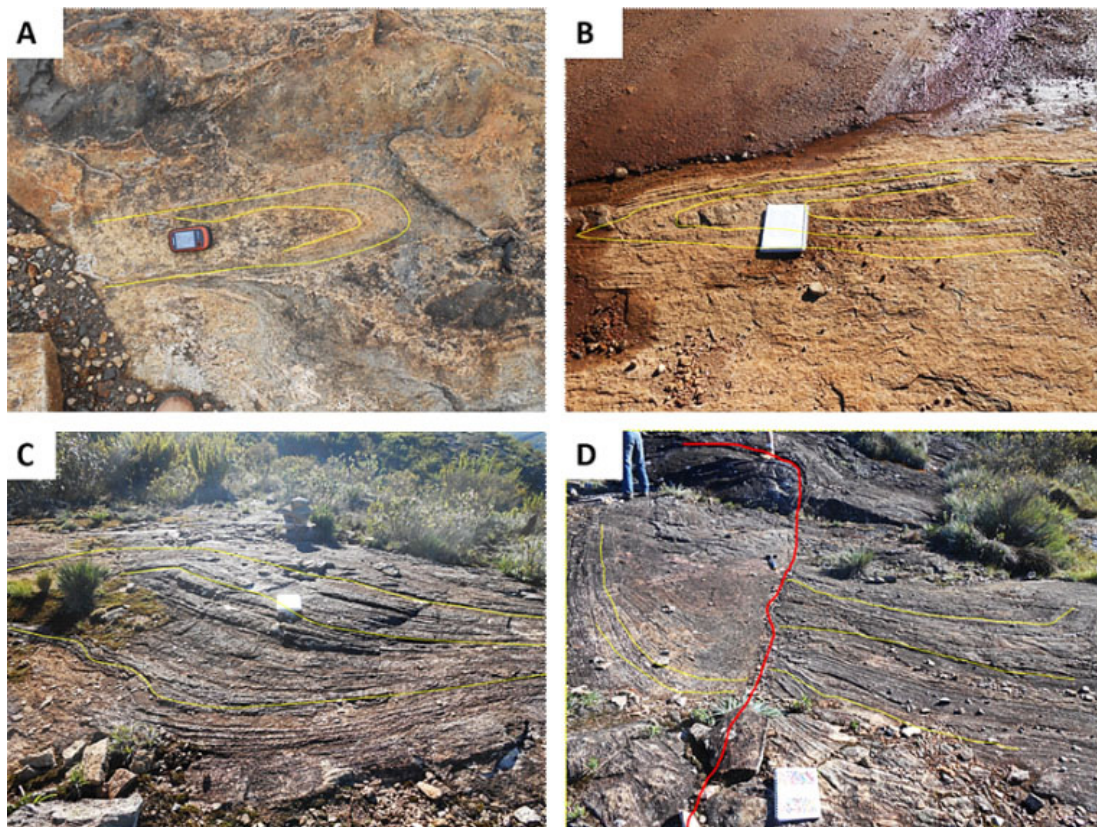


FIGURE 5. Images of outcrops with complex folded features.

TABLE 1. Rock samples and composition

Rock type	samples	description
Granulite	CPR03, CPR04, CPR08, CPR10	Quartz, K-Feldspar, Biotite, and pyroxene
Migmatite	CPR13, CPR18, CPR 20	Quartz, K-Feldspar, Biotite, and amphibole

TABLE 2. Configuration of Faraday and CDD collectors for U-Pb isotopic analyzes.

H4	H3	H2	H1	C	L1	L2	L3	L4	IC1	IC2	IC3	IC4	IC5
²³⁸ U		²³² Th											
									²⁰⁸ Pb	²⁰⁷ Pb	²⁰⁶ Pb	²⁰⁴ Pb	²⁰² Hg

TABLE 3. Operating conditions of LA-ICP-MC-MS in the U-Pb method.

ICP-MC-MS – NEPTUNE PLUS (Termo Scientific)	
RF Energy:	1200W
Gas flow:	Ar Coll: 15L/min Ar Aux: 0,73 L/min Ar carrier: 0,85 L/min
Analysis Mode:	Static
Detectors:	Faradays + ion counting
Acquisition time:	1,045s
Laser ablation – Machines In. 193 mm	
Crater diameter:	30 μm
Laser pulse energy:	4-7 j/cm ²
Frequency:	6 Hz
Abration time	40 s
Gas flow (He) (A):	0,80 L/min
Gas flow (He) (B):	0,22 L/min

He gases, being measured as a background, followed by the measurement of a shot made on a reference material zircon (GJ-1). If the data are compatible with the true value reported in the literature (Elhoul et al. 2006; Jackson et al. 2004). that is, for $^{206}\text{Pb}/^{207}\text{Pb} = 0.06389$ and $^{206}\text{Pb}/^{238}\text{U} = 0.09812$, so the analysis of nine grains of unknown age can be started. The choice of the grain to be analyzed and the crater position was made using the cathodoluminescence images and the image provided by the equipment camera. The laser was positioned at the chosen location and the beam was manually activated, initiating the ablation process; then data acquisition was initiated in 40 cycles. With the completion of the unknown analyzes, data of another reference material (91500) was obtained, ending the spreadsheet with the reading of a second GJ-1 and a blank again. The U-Pb results obtained by LA-ICP-MS were treated in an offline spreadsheet for blank and GJ-01 correction. Finally, the program ISOPLOT (version 4.1.5 of Ludwig 2003) was used to calculate ages and construct concordia diagrams. The decay constant values used in this work were the ones recommended by Steiger and Jäger (1977). All values were referred to as 2σ for all the uncertainties cited in the body of the text.

The data obtained in the mass spectrometer was processed using an Excel spreadsheet, where off-line corrections were performed through the blanket procedure, following the sequence: blank, GJ1 zircon reference material, nine unknown analyzes, the 91500 and GJ1 reference material, and one blank again. The procedure works to correct the average of the final blank and for reference materials.

The abundance determination of the U-Pb isotopes in the zircon grains consisted of reading isotope concentrations where the material volatilization procedure occurs (crater)

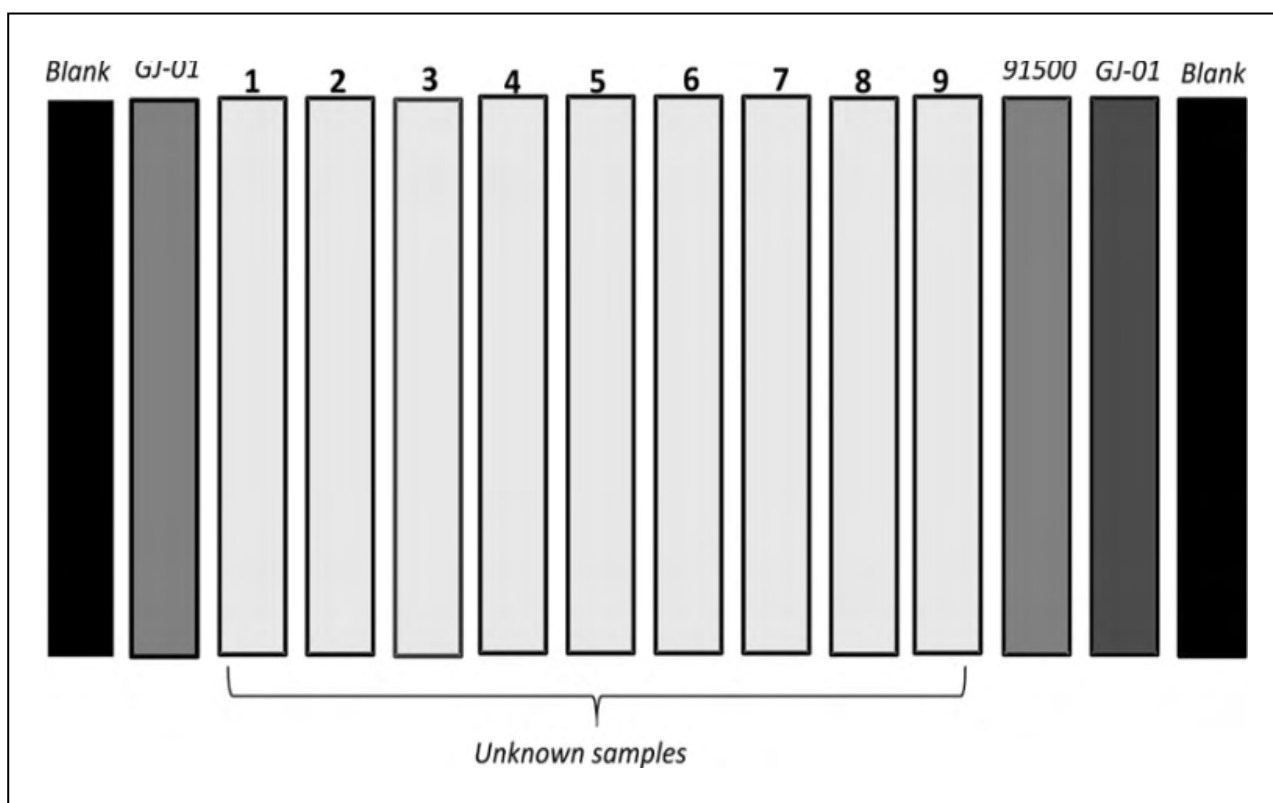


FIGURE 6. Schematically bracketing procedure used for reference material and blank corrections.

through the laser ablation (LA) method. In the isotopic data collection, the sample mount was inserted in the reserved compartment inside the device, together with the reference material. In the U-Pb method, GJ-1 and 91500 were used. Isotopic data were obtained using the static mode through 40 cycles of 1.054 seconds acquirement time with a gas inlet flow (Ar) of 15 L/min, auxiliary flow (Ar) 0.8 L / min, in the MC-ICP-MS.

Data on the GJ-1 zircon standard for a 30µm ablation spot usually yielded 432,000-114,000 cps of ^{206}Pb , 25,000-7,000 cps of ^{207}Pb , 6,500-4,200 cps of ^{208}Pb , 4,400-4,200 cps of ^{202}Hg and 1,060-1,090 cps of $^{204}(\text{Hg}+\text{Pb})$. For ^{232}Th and ^{238}U , measurements on faraday cups the values are 0.78 mV and 6.06 mV, respectively, yielding an age of 610.8 ± 2.6 Ma (Table 4; Figure 7). The GJ-01 reference material comprises a large amount of zircon crystals approximately 1 cm in size from African pegmatites (Elhlou et al. 2006; Morel et al. 2008) with crystallization age of 608.5 ± 0.4 Ma (Jackson et al. 2004). Using an Excel spreadsheet, off-line corrections for blank, Hg interference and common lead were performed. In addition, the obtained GJ-1 reference material values (Table 5) were compared with the literature values (Elhlou et al. 2006). Hence, the U-Pb results obtained by LA-ICP-MS were treated in an off-line spreadsheet for blank and GJ-1 correction. A second reference material was used, the 91500 (Figure 8), which represents a single crystal of zircon from a syenite pegmatite from the Renfrew County mine, Canada, crystallized at 1065 ± 6 Ma (Griffin et al. 2006).

4.3. Lu-Hf isotopes

The Lu-Hf isotopic system is one of the most innovative and powerful tools in zircon geochronology (e.g., Amelin et al. 1999; Griffin et al. 2000, 2002; Woodhead et al. 2004; Belousova et al. 2006; Gerdes and Zeh 2006, 2009; Hawkesworth and Kemp 2006; Zeh et al. 2007). The Lu-Hf technique is applied to zircon grains because this mineral has high Hf concentration, because it replaces Zr, and preserves the initial ratios of Hf. The blocking temperature of the Hf in the zircon is about 200 °C higher than the Pb (approximately 1,100 °C), indicating that the Hf isotopic system is closed during almost all thermal events, such as high-grade metamorphism, maintaining the isotopic ratios present in the zircon crystallization (Duchene et al. 1997; Choi et al. 2006; Schmidt et al. 2008). The models of Hf isotopic evolution have been proposed based on the hypothesis of the use of the Hafnium as a marker of the geochemical differentiation between the mantle and the crust (Patchett et al. 1981; Amelin et al. 1999; Vervoort and Blichert-Toft 1999; Hawkesworth and Kemp 2006). In this sense, interpretations of ϵHf values are similar to those of ϵNd values being able to indicate mantle-derived rocks or rocks originating from crustal magmas (if the values are positive or negative, respectively). It has been widely used for understanding crustal evolution and mantle/crust differentiation.

The acquisition of Lu-Hf data is carried out following a sequence that begins with the choice of the grain to be analyzed and the place where the crater will be made, usually in the place where the U-Pb age was performed, however with a diameter of 40-50µm. For this, the CL images and the image provided by the equipment camera were considered when choosing the location to analyze, then the laser spot was

positioned, and the beam was manually activated, initiating the ablation process. The material ablated by laser was carried out using Ar and He gases and it took a few seconds (3-10 s) for the signal to stabilize. In sequence, data acquisition were obtained in 40 cycles (1.045 seconds each cycle).

The collectors were positioned as follows (Table 6): mass ^{176}Hf in the central collector, mass ^{177}Hf in the collector H1, mass ^{178}Hf in H2 and mass ^{179}Hf in H3. In the collectors L1 the mass ^{175}Lu , L2 the mass ^{174}Hf , L3 the mass ^{173}Yb and in L4 the mass ^{171}Yb . The isobaric corrections were installed in the mass spectrometer software, so that the interferences ^{176}Lu and ^{176}Yb had their abundances obtained through the measurements of the masses ^{173}Yb and ^{175}Lu . Thus, the correction factors of 0.795015 and 0.026580 were used, respectively. The correction of the isotopic fractionation of the mass spectrometer is performed from the constant ratios $^{179}\text{Hf}/^{177}\text{Hf}$ (true value 0.7325) and $^{171}\text{Yb}/^{173}\text{Yb}$ (true value of 1.123456) reported in the literature (Patchett and Tatsumoto 1980, 1981; Morel et al. 2008).

A calibration procedure of Faraday detectors (Table 7) was then performed using the reference material solution (JMC475) through plasma settings and gas (Ar) flows for signal optimization (Woodhead et al. 2004). Isotopic data were obtained using the static mode through 50 cycles of 1.054 seconds acquirement time with a gas inlet flow (Ar) of 15 L/min, auxiliary flow (Ar) 0.8 L/min, in MC-ICP-MS. The laser was connected with two input He streams with volumes of 0.800 l/m and 0.220 l/m, totalizing 1.020 l/m. The laser repetition was at 10 Hz, with 4-7 J / cm² (35-60%) output power and 40 µm crater size.

4.4 Lu-Hf Calibrations

The abundances values of the Hf, Lu and Yb isotopes were reliable according to the three reference materials used (GJ-1, Mud Tank and 91500). In the calibration of the Lu-Hf method using laser ablation, the $^{176}\text{Hf}/^{177}\text{Hf}$ ratios of the GJ-1 reference material were initially analyzed. The GJ-1 (Figure 9) is used in large scale by geochronology laboratories being a reference material for U-Pb and Lu-Hf isotopic analysis. The isotopic ratios $^{176}\text{Lu}/^{177}\text{Hf}$ and $^{176}\text{Hf}/^{177}\text{Hf}$ of this reference material were reported in the literature (Woodhead and Hergt 2005; Elhlou et al. 2006; Morel et al. 2008) with values of 0.00025 and 0.282005 ± 5 , respectively. In the calibration of the method using laser, the $^{176}\text{Hf}/^{177}\text{Hf}$ ratios of the GJ-1 were similar to the published reference material values. The average ratio value was 0.282016 ± 5 , which is almost identical to the recommended value in the literature.

A second reference material used during the analysis is comprised of Mud Tank (Figure 9), a natural zircon collected in a carbonatite that outcrops in Strangways, east of Alice Springs (Australia). This carbonatite has an age of 732 Ma and has large amounts of zircon and apatite crystals up to ten centimeters. The obtained isotopic ratios $^{176}\text{Lu}/^{177}\text{Hf}$ and $^{176}\text{Hf}/^{177}\text{Hf}$ were 0.000042 ± 6 and 0.2882507 ± 6 , respectively, which are equivalent to those described in the literature for the Mud Tank reference material (Woodhead and Hergt 2005).

The 91500 (Figure 9) reference material was also used. It was part of the Harvard Museum collection and was carefully prepared as a reference material after a preliminary characterization, including Lu-Hf isotopic analyzes. Zircon 91500 has been widely adopted by many laboratories as

TABLE 4. LA-ICP-MC-MS in the U-Pb results of GJ-1 reference material.

Spot number	<i>f</i> 206a	Pb ppm	Th ppm	U ppm	Th/Ub	²⁰⁷ Pb/ ²³⁵ U	1 s [%]	²⁰⁶ Pb/ ²³⁸ U	1 s [%]	Rhod	²⁰⁷ Pb/ ²⁰⁶ Pbe	1 s [%]	²⁰⁶ Pb/ ²³⁸ U	1 s abs	²⁰⁷ Pb/ ²³⁵ U	1 s abs	²⁰⁷ Pb/ ²⁰⁶ Pb	1 s abs	% Concord
003-Sample1	0.005477466	30.06282838	6.081669973	301.9533515	0.020141091	0.824007886	4.052433176	0.099335992	1.834997771	0.452813826	0.060162166	3.613170053	610.5163828	11.20296201	610.2819872	24.73126972	609.4120401	22.01909333	100.1812144
003-Sample1	0.004144864	31.32510126	6.141735653	316.2858571	0.019418306	0.823796581	3.371622299	0.099165922	1.785387642	0.529533703	0.06024989	2.86010977	609.5190274	10.88227739	610.1643517	20.57243735	612.5609202	17.51991473	99.50341383
004-Sample 2	0.005425667	31.87489874	6.458264347	322.9166958	0.019999785	0.816388279	3.39337087	0.098647078	1.764412704	0.519958699	0.06002211	2.898588221	606.4753848	10.70072873	606.0314438	20.56489448	604.3717471	17.51824827	100.3480702
003-Sample1	0.000723882	33.49376938	6.92654484	339.2692516	0.02041607	0.819742823	2.369111315	0.098681867	1.347150792	0.568631277	0.060247495	1.948813271	606.6795053	8.172887761	607.9049449	14.40194484	612.4750257	11.93599459	99.05375401
004-Sample 2	0.001065214	29.70623062	5.67345516	299.9333013	0.018915723	0.820426983	2.826345368	0.099131133	1.734536616	0.613702994	0.060024505	2.231504172	609.3149967	10.56879173	608.2866219	17.19228077	604.4580811	13.4885073	100.8035157
003-Sample1	0.002170495	31.6	6.3	319.6012765	0.019712061	0.820088356	2.440273061	0.0989065	1.410143996	0.577863198	0.060136	1.991588945	607.9973857	8.573638632	608.0977279	14.83924504	608.4716136	12.11825339	99.92206245
003-Sample1	0.003769035	29.03815828	5.728594541	289.5010172	0.019787822	0.844114448	3.417973185	0.101450121	1.359496298	0.397749258	0.060345861	3.135970426	622.9015515	8.46832353	621.4136046	21.23975037	615.9986906	19.31753676	101.1205967
004-Sample 2	0.003769035	29.26287264	5.703050476	289.1737738	0.01972188	0.846452268	3.393735036	0.101661395	1.287540263	0.379387386	0.060387233	3.140012351	624.1379522	8.036027429	622.7000101	21.13278841	617.478364	19.38889689	101.0785136
ARI 17 (D) 01	0.002360413	31.92863133	6.417043187	325.7854435	0.019697145	0.818000087	2.307614603	0.098652597	1.192795377	0.516895402	0.060137306	1.975430167	606.5077648	7.23439658	606.9325015	14.00566303	608.5185664	12.02085933	99.6695579
ARI 17 (C) 01	0.002379237	32.21276906	6.405464223	324.3234968	0.019750232	0.830222278	2.467139778	0.100146822	1.220815209	0.494830175	0.060125117	2.143919054	615.2692669	7.511300785	613.7355157	15.14171304	608.0803156	13.03674975	101.1822371
ARI 17 (C) 02	0.002018368	30.98723094	6.194535777	314.8790561	0.019672746	0.809950713	2.619744015	0.097666178	1.321742064	0.504530999	0.060146883	2.26187016	600.7172922	7.939933135	602.4263739	15.78202887	608.8628152	13.77168633	98.66217433
ARI 17 (B) 02	0.003178273	40.1785558	7.972566628	404.2939044	0.01971973	0.828293715	2.682013354	0.100512535	1.27502357	0.47539792	0.059767194	2.359557274	617.4118448	7.872146544	612.6650104	16.43175739	595.1565998	14.04306084	103.7393931
ARI 17 (A) 01	0.004986568	24.32898541	4.896934876	248.9959986	0.019666721	0.810258523	3.292932808	0.097041422	1.510095343	0.458586746	0.060557116	2.926263579	597.0471561	9.015981298	602.5990412	19.84318153	623.5398328	18.24641903	95.7512455
ARI 17 (A) 02	0.004139965	38.87101459	7.703065124	390.2065543	0.019740994	0.829701603	3.004318594	0.100771578	1.397889975	0.46529352	0.059714884	2.65929198	618.9290462	8.651947091	613.4466115	18.42989062	593.2590141	15.77648938	104.3269519
CA-14-A1	0.002650275	38.45949184	9.406424903	385.8370959	0.024379265	0.830639157	2.403451352	0.099624575	1.605275523	0.667904313	0.060470651	1.788761833	612.2083858	9.827631366	613.9667685	14.7563926	620.4576424	11.0985095	98.67045612
CA-14-A2	0.002694392	24.74050816	3.193575097	253.365457	0.012604619	0.809603822	3.184728489	0.098188425	2.399140715	0.753326609	0.059801349	2.094425788	603.7836331	14.48561897	602.2317494	19.17944609	596.3944081	12.49103828	101.2389829
CA-1-(D) 1	0.002461032	23.41418277	4.962662571	242.0716777	0.020500798	0.813095378	2.119367114	0.098089688	1.031848792	0.486866473	0.060119708	1.851217177	603.2040136	6.224153327	604.1890015	12.804983	607.8857882	11.25328613	99.2298266
CA-1-(D) 2	0.001471668	39.78581723	7.637337429	397.1308752	0.019231286	0.827085004	1.852878085	0.099723312	1.196522318	0.645764191	0.060152292	1.414740803	612.7871963	7.332135566	611.9935046	11.33949353	609.0572229	8.616581047	100.6124176
CA-1-(C) 2	0.003336702	30.25869307	5.503206232	301.4031778	0.01825862	0.840478448	2.730154254	0.101107024	1.591227807	0.582834397	0.060289819	2.218498662	620.8932072	9.879825364	619.4096199	16.91083808	613.9921215	13.621407	101.1239697

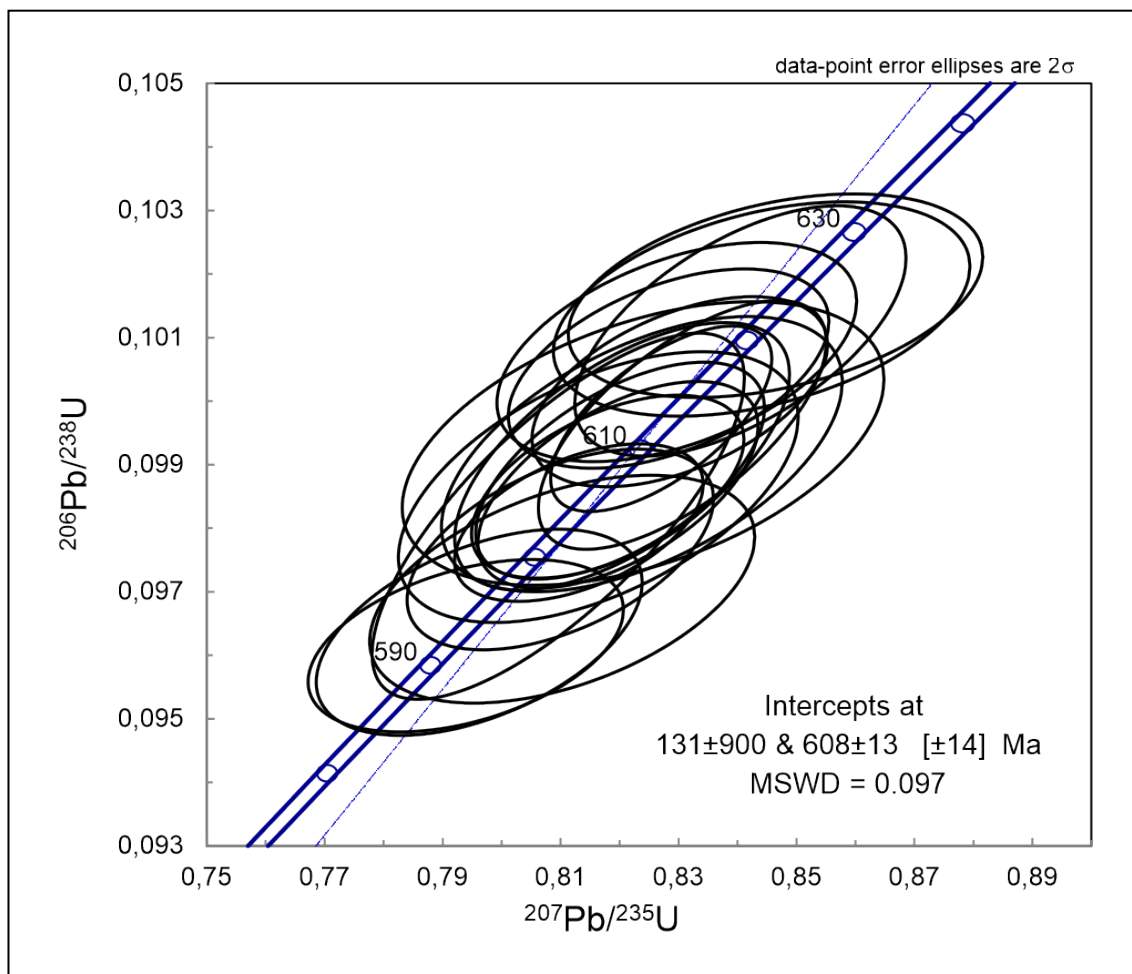


FIGURE 7. Concordia diagram of GJ-1 (reference material) U-Pb isotopic results obtained in the MultiLab laboratory during the analysis of unknown samples reported herein.

reference material for Lu-Hf analyzes. The isotopic ratios values of 0.000311 ± 8 ($^{176}\text{Lu}/^{177}\text{Hf}$) and 0.2882305 ± 8 ($^{176}\text{Hf}/^{177}\text{Hf}$) were determined and are consistent with the reported values in the literature (Elhlou et al. 2006; Morel et al. 2008).

The data were recorded by computers that are coupled to the mass spectrometer and then transferred to another computer for processing in Excel where the obtained GJ-1 values were compared with the literature values (Elhlou et al. 2006; Morel et al. 2008) to calculate T_{DM} and ϵ_{Hf} values. Finally, the Excel spreadsheet was used to make the Hf isotopic evolution diagrams. The procedure was used in order to correct the average of the final blank and for reference materials (detailed procedures may be found in Alves et al. 2019). T_{DM} ages and ϵ_{Hf} were calculated assuming an average crustal $^{176}\text{Lu}/^{177}\text{Hf}$ value of 0.01250 (Patchett et al. 1981; Griffin et al. 2000; Chauvel et al. 2014), using an offline Excel spreadsheet (Bertotti et al. 2013). In all the tables and figures, the initial ϵ_{Hf} was calculated using present-day CHUR values of $^{176}\text{Hf}/^{177}\text{Hf} = 0.282785$ and $^{176}\text{Lu}/^{177}\text{Hf} = 0.0336$ (Bouvier et al. 2008). For diagrams of the crustal and depleted mantle evolution curves (Patchett and Tatsumoto 1980; Vervoort and Blichert-Toft 1999), the present-day values for $^{176}\text{Hf}/^{177}\text{Hf}$ in the depleted mantle and Archean crust are 0.283214 and 0.280554, respectively, and corresponding values for $^{176}\text{Lu}/^{177}\text{Hf}$ are 0.0399 and 0.0024.

For all the samples, the U-Pb method was performed and crystallization ages were calculated prior to the Lu-Hf

analyses. This procedure was necessary because for the calculations of the ϵ_{Hf} parameter it is necessary to obtain the crystallization ages. In this sense, 10 zircon grains of each sample were chosen for the analysis; the laser crater of the Lu-Hf analysis was positioned in the same area of the crater produced by the U-Pb method.

5. Results and data analyses

5.1. Banded felsic granulite (Sample CPR 03)

The zircon grains occur with morphology varying from prismatic to elongate and show the length/width ratio ranging from 1:1 to 1:5. In general, the grains show rounded endings and in rare cases pyramidal endings. The cathodoluminescence (CL) images show well-defined oscillatory zoning (with layers varying in thickness), a typical growth texture in magmatic processes (Figure 10A).

For this sample, a total of 19 grains were analyzed, which generated the concordia diagram (Figure 11A). Most of the analyses are concordant, with discordance values <10%. The analyses exhibit Th/U ratios ranging from 0.5 to 1.5 (Table 8), indicating that the zircon grains were derived from magmatic rocks.

For the construction of the concordia diagram and age calculation, the fifteen measurements with the lowest error and the most concordant results were selected. The age

TABLE 5. LA-ICP-MS in the U-Pb results of 91500 reference material

Spot number	f 206a	Pb ppm	Th ppm	U ppm	Th/Ub	Isotope ratios									Ages (Ma)				
						²⁰⁷ Pb/ ²³⁸ U	1 s [%]	²⁰⁶ Pb/ ²³⁸ U	1 s [%]	Rhod	²⁰⁷ Pb/ ²⁰⁸ Pb	1 s [%]	²⁰⁶ Pb/ ²³⁸ U	1 s abs	²⁰⁷ Pb/ ²³⁸ U	1 s bs	²⁰⁷ Pb/ ²⁰⁸ Pb	1 s abs	% Concord
012-91500	0.009961	8.792298	4.853542	46.54754	0.104271	1.835211	4.213146	0.176322	2.739343	0.650189	0.075488	3.201031	1046.848	28.67676	1058.147	44.5813	1081.53	34.62012	96.79322984
012-91500	0.010988	8.596215	-26.8882	42.96674	-0.62579	1.947319	6.306179	0.1874	2.905876	0.460798	0.075364	5.596765	1107.277	32.17609	1097.523	69.21178	1078.233	60.34614	102.6936837
012-91500	0.00739	8.071079	1.06961	39.96361	0.026765	1.890144	3.552354	0.1821	2.849466	0.802135	0.075281	2.121263	1078.436	30.72966	1077.633	38.28132	1076.009	22.82497	100.2255462
012-91500	0.008758	8.399928	6.302696	40.96072	0.153872	1.913471	5.487691	0.183427	3.707488	0.675601	0.075659	4.045897	1085.668	40.251	1085.795	59.58507	1086.05	43.94047	99.96478502
012-91500	0.021059	5.955597	-7.68195	29.2964	-0.26221	1.942041	6.969953	0.187465	5.828175	0.836186	0.075134	3.822646	1107.626	64.55439	1095.704	76.37003	1072.094	40.98237	103.3142659
012-91500	0.009631	6.485514	6.235973	33.27502	0.187407	1.89662	4.96548	0.183421	3.476582	0.70015	0.074994	3.54533	1085.638	37.74311	1079.905	53.62247	1068.354	37.87666	101.6179081
012-91500	0.018527	5.966631	118.018	29.74039	3.968275	1.946318	6.750591	0.185945	5.342696	0.791441	0.075915	4.126266	1099.372	58.73611	1097.178	74.06603	1092.83	45.09306	100.5986556
012-91500	0.014414	6.582439	4.217703	31.60079	0.133468	2.010495	6.354589	0.193272	4.761473	0.749297	0.075445	4.208228	1139.076	54.2368	1119.058	71.11157	1080.392	45.46534	105.4317949
012-91500	0.013271	7.261859	4.198266	36.17678	0.116049	1.917528	6.151084	0.183603	3.87228	0.629528	0.075746	4.779255	1086.63	42.07737	1087.208	66.87507	1088.365	52.01576	99.84056218
012-91500	0.019002	5.969732	3.916536	29.67938	0.131961	1.954757	6.659129	0.186735	5.45709	0.81949	0.075922	3.816302	1103.666	60.22802	1100.083	73.25593	1093.003	41.71229	100.9755336
012-91500	0.002682	9.441448	19.663	44.92839	0.437652	1.922687	4.818485	0.18475	4.190815	0.869737	0.075479	2.377996	1092.87	45.80015	1089.002	52.47339	1081.275	25.71267	101.0723461
012-91500	0.025914	4.625964	10.51357	21.27085	0.494271	2.104767	9.327857	0.199696	7.766245	0.832586	0.076442	5.166659	1173.689	91.15158	1150.367	107.3046	1106.671	57.17794	106.0558042
012-91500	0.003693	11.4477	18.6438	55.76288	0.334341	1.929789	4.947299	0.184519	4.546394	0.918965	0.075852	1.950915	1091.617	49.62921	1091.466	53.99809	1091.165	21.2877	100.0414389
012-91500	0.00283	10.546	22.49904	52.06992	0.432093	1.908628	3.115533	0.183301	2.658026	0.853153	0.075519	1.625253	1084.985	28.83917	1084.106	33.77567	1082.339	17.59075	100.2444196

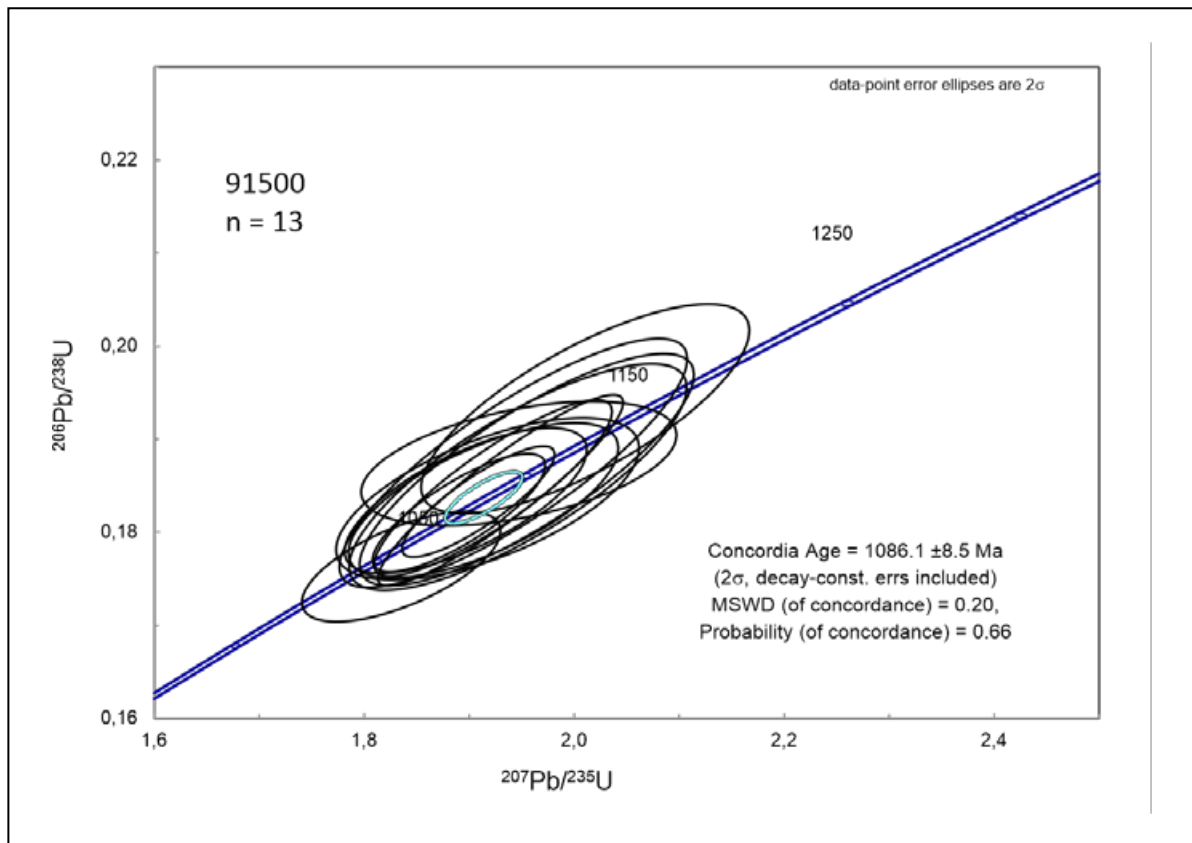


FIGURE 8. Concordia diagram of 91500 (reference material) U-Pb isotopic results obtained in the laboratory during the analysis of unknown samples reported herein.

TABLE 6 - Configuration of the Faraday collectors used for the Lu and Hf analyzes.

Faraday collectors	H3	H2	H1	C	L1	L2	L3	L4
Isotopes	^{179}Hf	^{178}Hf	^{177}Hf	^{176}Hf	^{175}Lu	^{174}Hf	^{173}Yb	^{171}Yb
Interferers				$^{176}(\text{Yb}+\text{Lu})$		^{174}Yb		

TABLE 7. Operating conditions of the LA-MC-ICP-MS for the Lu-Hf method.

ICP-MC-MS – NEPTUNE PLUS (Termo Scientific)	
RF Energy	1200W
Gas flow:	Ar Coll: 15L/min Ar Aux: 0,73 L/min Ar carrier: 0,85 L/min
Analysis Mode:	Static
Detectors:	Faradys
Aquisition time:	1,045s
Laser ablation – Machines In. 193 mm	
Crater diameter:	40 - 50 μm
Laser pulse energy:	4-7 j/cm^2
Frequency:	10 Hz
Abration time	50 s
Gas flow (He) (A):	0,80 L/min
Gas flow (He) (B):	0,22 L/min

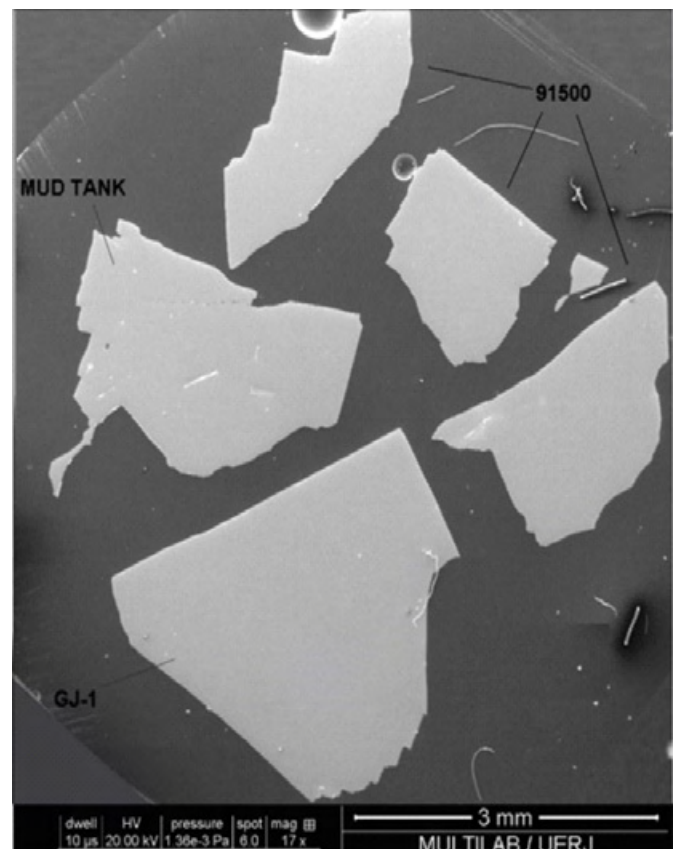


FIGURE 9. SEM images of the reference materials used for calibration of the Lu-Hf method in LA-ICP-MS in the MultiLab laboratory (UERJ).

obtained at the upper intercept of 2209 ± 22 Ma (MSWD = 5.00) is interpreted as the crystallization age of the magmatic orthogneiss protolith because the analyses correspond to the nucleus of the zircon grains. The CL images also show zircon grains with core and rims, and the analysis performed in some grains result in different ages, suggesting an isotopic re-homogenization process of the oldest nucleus during the growth of the edge in the reworking event (metamorphism in granulite facies). In cases where the core is light gray,

the best Paleoproterozoic ages were obtained, suggesting the preservation of the original isotopic composition. The Neoproterozoic zircon grains are light gray or dark gray in color and do not show very light oscillatory zoning but tend to have complex gray color variation patterns. Neoproterozoic ages were obtained in white rims but in several cases all the grain was completely re-homogenized.

The results obtained in the white border of the grains yielded an age of 596 ± 19 Ma (obtained in the lower

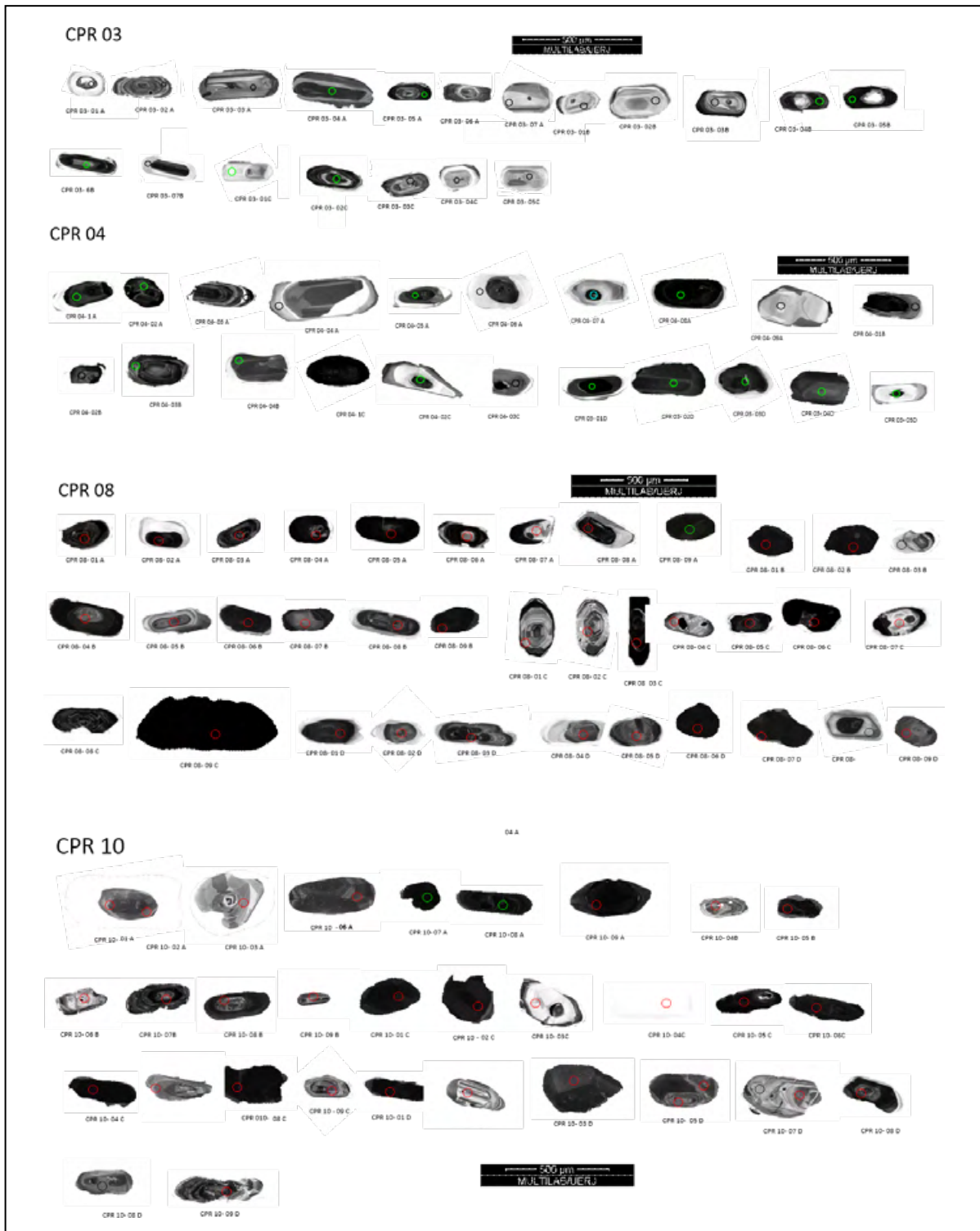


FIGURE 10. Cathodoluminescence images of the zircon grains of samples (A) CPR-3; (B) CPR 04; (C) CPR 08; and (D) CPR 10.

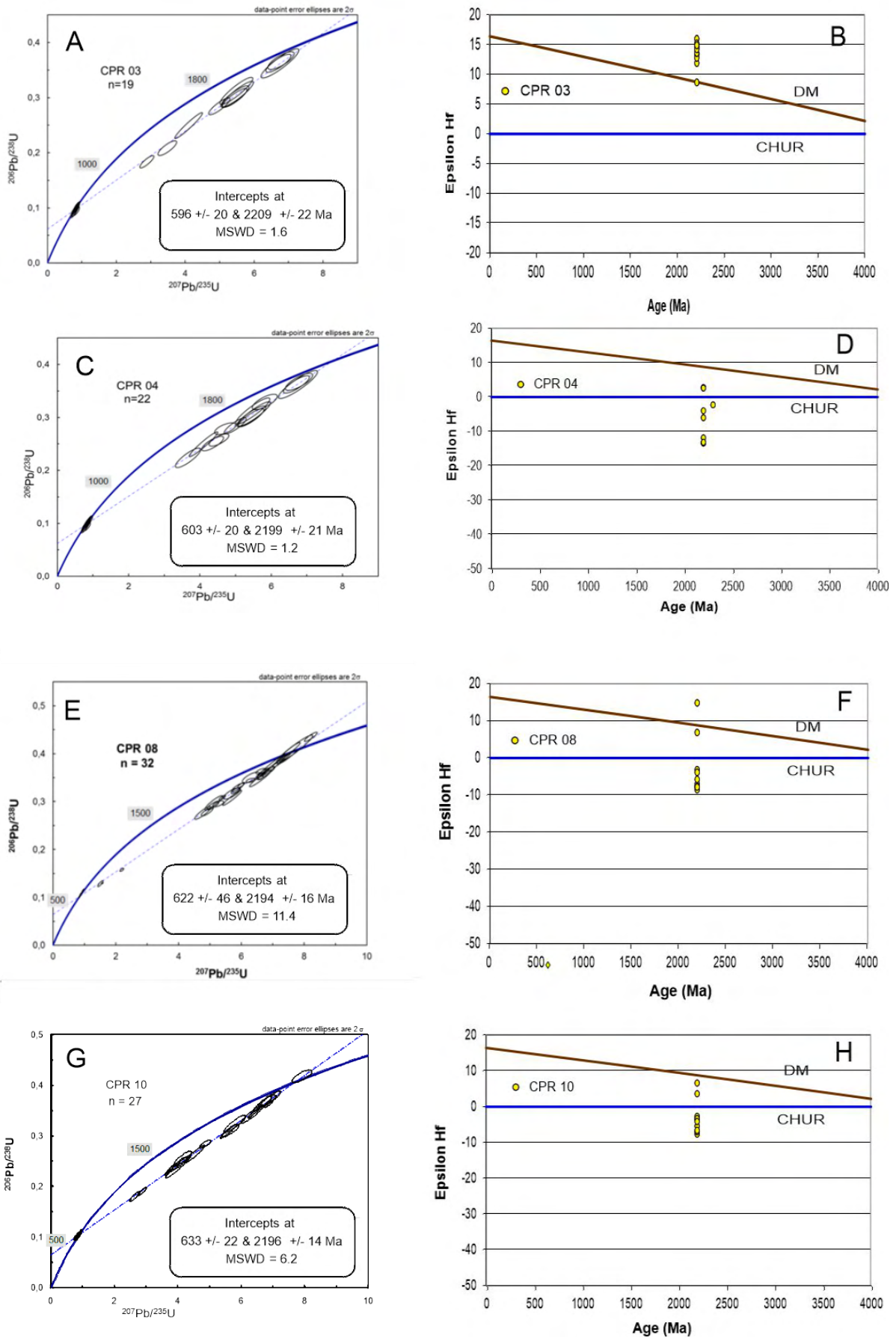


FIGURE 11. Cathodoluminescence images of the zircon grains of samples (A) CPR-3; (B) CPR 04; (C) CPR 08; and (D) CPR 10.

Table 9. Lu-Hf isotopes results obtained by LA-ICP-MS for the zircon grains of samples studied herein. (continued)

CPR-03	U-Pb Age (Ma)	±2s	Sample (Present day ratios)				Chur	DM	Sample Initial Ratios					DM Model Ages (Ga)				
			176Hf/177Hf	±2SE	176Lu/177Hf	±2SE	176Hf/177Hf (t)	176Hf/177Hf (t)	176Hf/177Hf (t)	εHf(0)	εHf(t)	±2SE	T DM	T DM Crustald	T DM Crustale	Mafic	Felsic	
010 Sample 7	2060	0.6	0.281451031	3.95783E-05	0.000263603	1.53402E-05	0.281467565	0.281728677	0.281440695	-47.1725467	-0.954633839	0.055966592	2.443785638	2.704211433	2.643306779	2.97036275	2.592923535	
011 Sample 8	2060	0.6	0.281421153	3.35803E-05	0.000621574	5.16923E-06	0.281467565	0.281728677	0.281396782	-48.22910319	-2.514800884	0.021946511	2.506195144	2.801768597	2.731698457	3.107832437	2.673724015	
012 Sample 9	2060	0.6	0.281418813	3.57438E-05	0.000530053	1.28026E-05	0.281467565	0.281728677	0.28139803	-48.31186207	-2.470454939	0.060703537	2.503465355	2.798998099	2.729188039	3.103929887	2.671429037	
013 Sample 10	2060	0.6	0.281408005	3.28051E-05	0.000821954	1.77502E-05	0.281467565	0.281728677	0.281375777	-48.69405251	-3.261062122	0.071753221	2.536800546	2.848369524	2.773926567	3.173462591	2.712329558	
014-91500	1065	0.6	0.282132729	3.22135E-05	0.000313373	3.93688E-07	0.282110227	0.282470797	0.282126435	-23.06597582	0.574561502	0.001111116	1.532765	1.834432585	1.761766168	2.151795672	1.701640961	
CPR-20	U-Pb Age (Ma)	±2s	176Hf/177Hf	±2SE	176Lu/177Hf	±2SE	176Hf/177Hf (t)	176Hf/177Hf (t)	176Hf/177Hf (t)	εHf(0)	εHf(t)	±2SE	T DM	T DM Crustald	T DM Crustale	Mafic	Felsic	
003-Mud Tank	732	5	0.28228663	2.59518E-05	3.77148E-05	1.79865E-07	0.282322656	0.282716102	0.282286111	-17.62364633	-1.294432378	0.015133999	1.314915363	1.691057368	1.600627258	2.085651184	1.52578143	
004 sample 1	629	0.6	0.281677945	5.51067E-05	4.2113E-05	1.30929E-06	0.282388095	0.282791669	0.281677448	-39.14828597	-25.16561722	0.811326561	2.129614104	3.079626401	2.851440484	4.068528393	2.662133786	
005 Sample 2	629	0.6	0.281455147	4.19481E-05	0.001072329	0.0001115	0.282388095	0.282791669	0.28144248	-47.02699084	-33.48634848	3.518805617	2.489390865	3.58240446	3.308563501	4.766492214	3.081203028	
006 sample 5	629	0.6	0.28140892	4.46768E-05	0.000427124	0.000133065	0.282388095	0.282791669	0.281403874	-48.66172074	-34.85347833	10.89691127	2.510083293	3.664563381	3.38329891	4.88030554	3.149745267	
007 Sample 4	629	0.6	0.281383504	3.76244E-05	0.000942772	4.22658E-05	0.282388095	0.282791669	0.281372367	-49.56049012	-35.96921316	1.65167214	2.57775431	3.731521218	3.444214394	4.973011384	3.205618671	
008 sample 5	629	0.6	0.281564771	4.26492E-05	0.000565759	3.89962E-05	0.282388095	0.282791669	0.281558088	-43.15042013	-29.39242446	2.058425102	2.310268007	3.335619905	3.084139667	4.42422011	2.875424432	
009 Sample 6	629	0.6	0.281658923	3.12581E-05	4.7249E-05	2.42675E-06	0.282388095	0.282791669	0.281658365	-39.82095035	-25.84137553	1.354754466	2.155150954	3.120635467	2.888711089	4.125553264	2.696290593	
010 Sample 7	629	0.6	0.281425866	4.93526E-05	0.001052063	0.000103061	0.282388095	0.282791669	0.281413438	-48.06245143	-34.51478686	3.42008823	2.52773259	3.644221147	3.364793744	4.852132061	3.132772849	
011 Sample 8	629	0.6	0.28146028	5.60642E-05	0.000840651	6.03833E-05	0.282388095	0.282791669	0.281450349	-46.84549618	-33.20768475	2.423572492	2.467627457	3.565642435	3.29331729	4.743263806	3.067221212	
012 Sample 9	629	0.6	0.281484937	3.72791E-05	0.00044924	7.53279E-05	0.282388095	0.282791669	0.28147963	-45.97355432	-32.170785	5.429298214	2.410096596	3.503225306	3.236548428	4.656742975	3.015163197	
013 Sample 10	629	0.6	0.281523143	4.42087E-05	0.000735659	8.90823E-05	0.282388095	0.282791669	0.281514453	-44.62249288	-30.93763752	3.780662231	2.376423367	3.428900052	3.168956846	4.553664598	2.953186538	
014-91500	1065	0.6	0.28203358	4.44019E-05	0.000321407	1.10595E-06	0.282110227	0.282470797	0.282027126	-26.57212129	-2.945689415	0.012259277	1.667032426	2.054289822	1.961032857	2.461161253	1.883843615	

intercept). This age is interpreted as regional metamorphism. The lower intercept may result from episodic Pb loss due to heating (metamorphism or hydrothermal alteration) according to Wetherill (1956) or continuous Pb loss (Tilton 1960; Wasserburg 1963).

Figure 11B shows the Lu and Hf isotope analytical results (Table 9) of the zircon grains from sample CPR 03. Ten Lu-Hf analyses resulted in T_{DM} 'crustal' model age values ranging between 1.83 and 2.22 Ga and ϵ_{Hf} values ranging from 8.7 to 16, calculated for the U-Pb age of 2209 ± 22 Ma. These results indicate that the analyzed rock was generated from a magma whose epoch of mantle extraction is close to the U-Pb age suggesting a juvenile character for this sample.

5.2. Banded charno-enderbite (Sample CPR 04)

The zircon grains morphology of the sample CPR 04 shows a length to width ratio ranging from 1:1 to 1:5. CL images present grains with whitish rims involving dark gray cores (Figure 10B).

In the sample CPR 04 a total of 22 grains were used, and the Concordia diagram obtained for these analytical data (Table 8) is presented in Figure 11C. In the construction of the concordia diagram and age calculation, the selected data presented concordances ranging from 70 to 99% and errors smaller than 10.87 (%), which resulted in small ellipses. The age obtained in the upper intercept of 2199 ± 20 Ma (MSWD = 1.2) is interpreted as the age of crystallization of the orthogneiss protolith and the age 600 ± 20 Ma obtained in the lower intercept is interpreted as representing the metamorphic peak. When the core has a lighter gray color, the oscillatory zoning is observed, and the ages indicate Paleoproterozoic isotopic inheritance. The rims of light gray grains have Neoproterozoic ages.

The analytical results (Table 9) of the Lu and Hf isotopes of the sample CPR 04 are shown in Figure 11D. Ten Lu-Hf analyses resulted in T_{DM} 'crustal' model age values between 2.51 and 3.39 Ga and the ϵ_{Hf} values ranging from -14.3 to 2 calculated for the U-Pb age of 2109 ± 20 Ma. The Lu-Hf results show positive and negative ϵ_{Hf} values, indicating that the magmatic sources of the protolith are mantle-derived with important contribution from older crust.

5.3. Granitic gneiss (Sample CPR 08)

The cathodoluminescence images of the CPR 08 sample (Figure 10C) show that the zircon grains have in general rounded shapes; in this sample, subeuhedral, grains predominate (length /width ratio ranging from 1:2 to 1:3, more homogeneous compared to the other samples). The oscillatory zoning pattern predominates and a small region with contrast in the luminescence at the center of some grains is common. Again, the metamorphic overgrowth is seen in the grains where the CL images show light gray shades.

A total of 32 spots on zircon grains were analyzed and the results are presented in Table 8 and are plotted in Figure 11E. The analytical errors are smaller than 4.6% and concordances vary between 76 and 110%. Concordia diagram plot described a clear discordance related to lead loss, yielding metamorphic age at the lower intercept of 622 ± 46 Ma. The magmatic crystallization age of the orthogneiss protolith is given by the upper intercept at 2194 ± 16 Ma, with MSWD of 11.4. The rims

of the zircon grains are thick enough and the analyzes carried out confirm the Neoproterozoic ages.

In Figure 11F the Lu and Hf isotopes analytical results (Table 9) of the sample CPR 08 are presented, which resulted in T_{DM} 'crustal' model age values between 1.88 and 3.06 and ϵ_{Hf} values ranging from -7, 8 and 14.8 calculated for the U-Pb age of 2194 ± 16 Ma.

The results indicate that the analyzed rock crystallized in the Paleoproterozoic was generated from a magma whose period of mantle extraction was in the Archean-Paleoproterozoic limit. In addition, the values of ϵ_{Hf} suggest a juvenile character for this sample, but with partial crustal contribution in its formation.

5.4. Granulitic gneiss (Sample CPR 10)

The zircons observed in sample CPR 10 mostly present the prismatic habit preserved (Figure 10D) but are rounding in the rims. The cores show growth with oscillatory zoning, characterized by the alternation of light and dark bands.

The U-Pb analyses (results presented in Table 8) of the sample CPR 10 are shown in Figure 11G, and they have analytical errors lower than 7.5 and concordant values ranging from 70% to 123% (MSDW = 6.2). For this sample, 27 spots were performed, which were concordant or plotted near the concordia curve, generating a population with a crystallization age of 2196 ± 14 Ma marking the upper intercept of the concordia curve and 633 ± 22 Ma, marking the inferior intercept related to the metamorphic event. The analyses in the zircon nucleus present Paleoproterozoic isotopic inheritance. Many grains have a fine metamorphic overgrowth indicated by the whitish color in the CL image. The analyses in this area of the grains were difficult due to the small size of the metamorphic border and because this region is fractured, which limits the quality of the analyses obtained. The results achieved in this portion of the zircon grains indicate Neoproterozoic age.

In Figure 11H, the Lu and Hf isotopes results of the studied zircon grains (reported in Table 9) are plotted. In the sample CPR 10, the ten Lu-Hf analyses resulted T_{DM} 'crustal' model age values ranging from 2.31 to 3.05 Ga and ϵ_{Hf} values ranging from -7.7 to 6.6 calculated for the U-Pb age of 2196 ± 14 Ma. As in sample CPR 03, the results of the sample CPR 10 indicate that the rock from which the zircons were extracted was generated from a mantle source, suggesting a juvenile character for this sample, however, there may have been some crustal contribution in the magmatic process (ϵ_{Hf} value reaches -7.7). The mantle extraction age of the sample yielded values from the Archean to the Paleoproterozoic. The negative ϵ_{Hf} values for the lower intercept age may be interpreted as result of the magma generation by anatexis process during the metamorphic peak, as indicated by petrographic studies.

5.5. Mylonitic granite (Sample CPR 13)

The cathodoluminescence images (Figure 12A) of the sample CPR 13 show a homogeneous family of magmatic, prismatic zircon crystals (ratio width/length ranging from 1: 2 to 1: 3), and euhedral to subhedral features. Most of these grains occur with oscillatory zoning and some with thin metamorphic overgrowth, with non-uniform zoning pattern. In this sample the difference between the oscillatory

magmatic pattern and the irregular pattern of metamorphic zoning is well characterized. The grains with irregular zoning pattern may be related to hydrothermal fluids associated with mylonitization.

For the sample CPR 13, nineteen zircon grains (Figure 13A) were analyzed for U-Pb (results in Table 10). After discarding the grains with discordant results and/or with high individual errors, a concordia diagram plot suggests magmatic crystallization age at 2060 ± 39 Ma (MSWD =

2.1). Concordance values are between 80 and 110%, but few grains can present values up to 150%. However, the errors assume values higher than in the other samples (up to 27% in some cases), but the ρ value for these spots remains close to 1. An age of 600 ± 18 Ma is observed in the lower intercept.

The Lu and Hf isotope results (Table 9) of the CPR 13 sample are shown in Figure 13B which resulted in T_{DM} 'crustal' model age values between 1.96 and 3.79 Ga and the



FIGURE 12. Cathodoluminescence images of the zircon grains of samples CPR-13, CPR 18 and CPR 20.

ϵ_{Hf} values range from -22.5 to + 13 calculated for the U-Pb age of $2092 \pm 68\text{Ma}$.

From the results it is possible to interpret the formation of this rock from the mixture of two sources (mantle and crust) in a period of mantle extraction that begins in the Archaean Eon going to the Paleoproterozoic Era, period when the rock was crystallized, as demonstrated by the U-Pb age. The metamorphic event that reaches this rock in the Neoproterozoic Era is characterized by crustal

reworking pointed out by the age of inferior intercept of the concordia diagram.

5.6. Granitic gneiss (Sample CPR 18)

From the CL images it was possible to individualize two groups of zircon grains in the sample CPR 18 (Figure 12B). The first of prismatic zircon crystals, elongated, with width / length pattern (1: 3 to 1: 4) and the second group of zircon

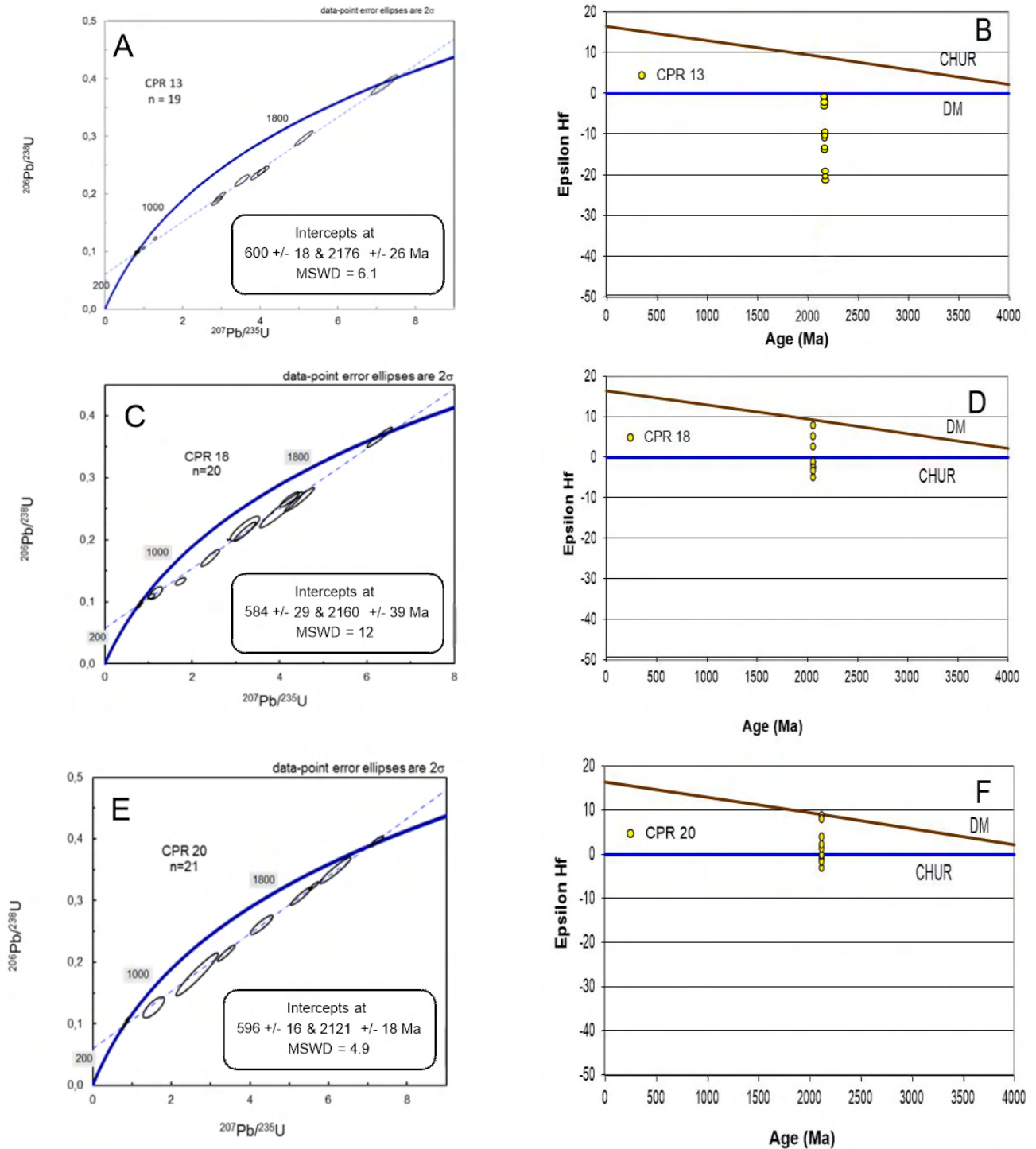


FIGURE 13. Concordia diagrams and Hf isotopic evolution for the zircon grains of samples (A) CPR-13, (B) CPR 18 and (C) CPR 20.

grains with rounded white ends. The zircon grains textures are oscillatory (boundaries between light and dark zones, straight and regular) or convolute (with boundaries between light and dark areas curved and irregular), but some regions present homogeneous light gray pattern.

The U-Pb results are presented in Table 10. In the concordia diagram it is observed that the spots with good analytical consistency are aligned in a straight discordia, characterizing events of episodic Pb loss (Figure 13C). This discordia line defines a superior intercept in 2060 ± 39 Ma, interpreted as age of magmatic crystallization, and a lower intercept in 584 ± 29 Ma, which indicates the Neoproterozoic metamorphism peak.

The analytical results (Table 9) of the Lu and Hf isotopes are shown in Figure 13D. Analyses resulted in T_{DM} 'crustal' model age values between 2.13 to 2.80 Ga and ϵ_{Hf} values ranging from -4.9 to +7.9 calculated for the U-Pb age of 2060 ± 39 Ma.

The results are consistent with the crystallization ages and metamorphism observed in the previous samples. The crystallization occurred in the Paleoproterozoic era from magmas originated from mixture of mantle and crustal sources and metamorphic ages point out to reworking of the rocks already formed in the Paleoproterozoic era.

5.7. Banded felsic protomylonitic gneiss (Sample CPR 20)

The sample CPR 20 shows zircon grains morphology (Figure 12C) ranging from well-formed crystals (1:4) to rounded crystals (1:1). Irregular zoning (interpreted as a result of metamorphic fluids or growth during the metamorphic event) predominates in some zircon families, and oscillatory zoning may also occur (in areas with preservation of the core with magmatic origin). Large number of nucleated grains are observed, with convolute zoning, homogeneous of low luminescence and oscillatory zoning. The analytical results of the Paleoproterozoic cores present a greater analytical error, probably due to the isotopic re-homogenization, while the rims grown during the metamorphic event present a more accurate analytical result with lower error and higher agreement.

The U/Pb analytical data (Table 10) obtained in the analysis of the sample CPR 20 are illustrated in Figure 13E with errors lower than 15% and concordant values ranging from 90 to 100%. The measurements of the 21 spots with better analytical coherence were selected, resulting in the concordance age of 2121 ± 18 Ma in the upper intercept, interpreted as the age of magmatic crystallization and 592 ± 16 Ma in the lower intercept, indicating the metamorphic event age.

Lu and Hf isotopes obtained for sample CPR 20, plotted in Figure 13F (results presented in Table 9), resulted in T_{DM} crustal model ages between 2.13 and 2.74 Ga and ϵ_{Hf} values ranging from -2.9 to 8.8 calculated for the U-Pb age of 2121 ± 18 Ma. Results for the U-Pb age of 596 ± 16 Ma, show ϵ_{Hf} values ranging from -36.0 to -25.2. Like sample CPR 13, the sample CPR 20 was crystallized in the Paleoproterozoic era and was formed from mantle and crustal sources, extracted from the mantle in the Archean eon to the Paleoproterozoic era. The metamorphism of this rock marks a period of crustal reworking evidenced by the negative ϵ_{Hf} values.

6. Discussion

Compositionally, the rocks of the Serra do Caparaó region are composed of an association of Paleoproterozoic enderbitic, charnockitic, dioritic, and gabbroic quartz granulites as reported in this work and data from literature (Silva et al. 2002). Some authors consider that the rocks of the Serra do Caparaó region are part of Juiz de Fora Complex (Seidensticker and Wiedmann 1992; Campos Neto and Figueiredo 1990). But such interpretation is not consensual and is still a matter of debate as noted by Noce et al. (2007), because some crystallization ages of charnockitic gneiss zircons have an age of 2195 ± 15 Ma, which is older than those found for Juiz de Fora Complex (2134-2084 Ma). Therefore, this work (i) discusses issues related to crystallization age and sources of Paleoproterozoic photolith and (ii) the Neoproterozoic metamorphic event and; (iii) propose a model for the evolution of the basement of the Ribeira-Araçuaí orogen.

6.1 The Ribeira-Araçuaí belt basement evolution (ages and sources)

The obtained U/Pb data allowed to identify two age groups in the studied rocks. The first group is associated with Paleoproterozoic magmatic rocks represented by the samples (CPR 03, CPR 04, CPR 08 and CPR 10) characterized by zircon grains with Paleoproterozoic nucleus and Neoproterozoic borders, interpreted as metamorphic ages. The second group of rocks is represented by samples CPR 13, CPR 18 and CPR 20 and presents Neoproterozoic ages. The ages obtained in most of the samples may be correlated with two thermal events reported in the literature, the oldest (see results in Tables 8 and 10) related to the Paleoproterozoic magmatic event from 2.2 Ga to 2.0 Ga Ma (Ávila et al. 2010) and a younger event linked to the isotopic re-homogenization of the Brasiliano Orogeny (Heilbron et al. 2004).

The group of ages ranging from 2209 ± 22 Ma to 2060 ± 39 Ma can be correlated to the implantation of the Juiz de Fora magmatic arc (as proposed by Heilbron et al. 2010; Ávila et al. 2010). It has the same age range of the rocks of the Serrinha and Ritapolis magmatic arcs (of about 2230 – 2180 Ma) and represents magmatic arcs originated by an oceanic lithosphere subduction related to the Paleoproterozoic grown of the São Francisco craton. Juiz de Fora magmatic arc developed after the consolidation of the Serrinha arc (very close to 2192 – 2121 Ma, reported to Ritapolis magmatic arc) in the Mineiro Belt, according to Ávila et al. (2010). Serrinha and Ritapolis present an intra-oceanic signature according to Ávila et al. (2006) and Juiz de Fora is marked by an important older continental crust participation in the magma generation (Noce et al. 1998).

The results obtained in this work suggest a possible regional correlation between the Caparaó complex and Juiz de Fora complex. The Paleoproterozoic ages determined in this work are similar to those obtained in granulitic rocks by U-Pb in zircons by other authors (Silva et al. 2002; Heilbron et al. 2004). It is also emphasized that the ages obtained in the above-mentioned works also present strong crustal reworking associated with the Brasiliano event.

The ϵ_{Hf} values results suggest that rocks crystallized from 2209 to 2060 Ma of the Caparaó Complex present juvenile (mantle source) magmatism with important continental

crust participation in the genesis of these rocks. Such a contamination would explain the variability of ϵ_{Hf} values from positive to negative (Figure 14).

The obtained T_{DM} 'crustal' ages from 2.13 to 3.87 Ga can be interpreted as the age of the mantle extraction of the studied rocks. Similar Lu-Hf model age has been reported in Paleoproterozoic crust (Teixeira et al. 2015; Martínez Dopico et al. 2017; Barbosa et al. 2018). The Neoproterozoic ages (633 to 583 Ma) that marked the lower intercept of some samples, corroborate the idea of crustal reworking occurred in the Brasiliano Orogeny related to the formation of the Ribeira – Araçuaí belts.

The U-Pb and Lu-Hf results reported here define two groups of ϵ_{Hf} values. The first group with positive ϵ_{Hf} values is correlated to rocks formed in magmatic arc. The second group indicates granulite facies metamorphism during paleoproterozoic rocks (from high amphibolite to granulite facies). In this way, the sin-collisional stage of the Mineiro Belt occurred during the process of plate convergence and compressive tectonics between opposite continental margins (São Francisco craton Archean core) and crustal duplication with increased pressure and temperature resulting in processes of metamorphism and anatexis. The collisional orogen was preceded by the pre-collisional stage, formed by the subduction of oceanic lithosphere, in which the calcium-alkaline magmatic arcs were built.

6.2 The Neoproterozoic metamorphic event time constraints

Metamorphism in the Ribeira-Araçuaí belts is associated with crustal thickening, corresponding to the M1 event represented by the collision of the Oriental Terrane with the Rio Negro magmatic arc (630-600 Ma), and with a later event M2, related to the collision (540-500 Ma) of the Costeiro Terrane and the Congo craton (Schmitt et al. 2004). The M1 event was responsible for the production of the main Ribeira Belt foliation and the intermediate

to high pressure paragenesis. In the Paleoproterozoic enderbite and charnockite orthogneisses of Juiz de Fora Complex, it was reported that the M1 geothermobarometric event was associated with pressures between 4-6 kbar and temperatures between 750°C and 850°C (Heilbron et al. 2004). This event is characterized by the infiltration of fluids rich in CO_2 , providing a granulitic metamorphism. According to Heilbron et al. (2004), the metamorphic degree of the Northern Ribeira Belt increases in the NW-SE direction, going from the greenschist facies to the granulite facies. In the NW area of the Ribeira Orogen, the Oriental Terrane, the metamorphism varies from the greenschist facies, on the edge of the São Francisco Craton, to the medium pressure granulite facies, close to the contact with the Oriental and Paraíba do Sul Terranes (700°C and 7 kbar).

A number of Neoproterozoic ages associated with the Brasiliano event were also recorded in the analyzed samples reported here. These ages are associated with metamorphic reworking processes and crystallization/generation of zircon grains. The obtained U/Pb ages can be associated with the time interval that the Rio Negro magmatic arc was developed in the Ribeira Orogeny, given by ages between ≈ 790 -620 Ma (Tupinambá et al. 1996, 2012).

6.3 Th/U ratios and zircon growth

The U/Pb method is an important tool for the investigation of metamorphic events since the isotopic results allow to characterize the discordance of the ages $^{235}\text{U}/^{207}\text{Pb}$ ages and $^{238}\text{U}/^{206}\text{Pb}$ ages, commonly attributed to the episodic loss of radiogenic Pb in an open system (Wetherill, 1956; Tilton 1960; Wasserburg 1963) caused by a thermal event. Thus, metamorphic growth of zircon from aqueous fluid versus anhydrous melt can be distinguished by the concentrations of Th and U. For example, a zircon grain crystallized with kyanite, garnet, titanite and quartz in granulite facies is interpreted as having grown from hydrothermal solutions and it has very low Th/U ratios from 0.003 to 0.068 (Zheng et al.,

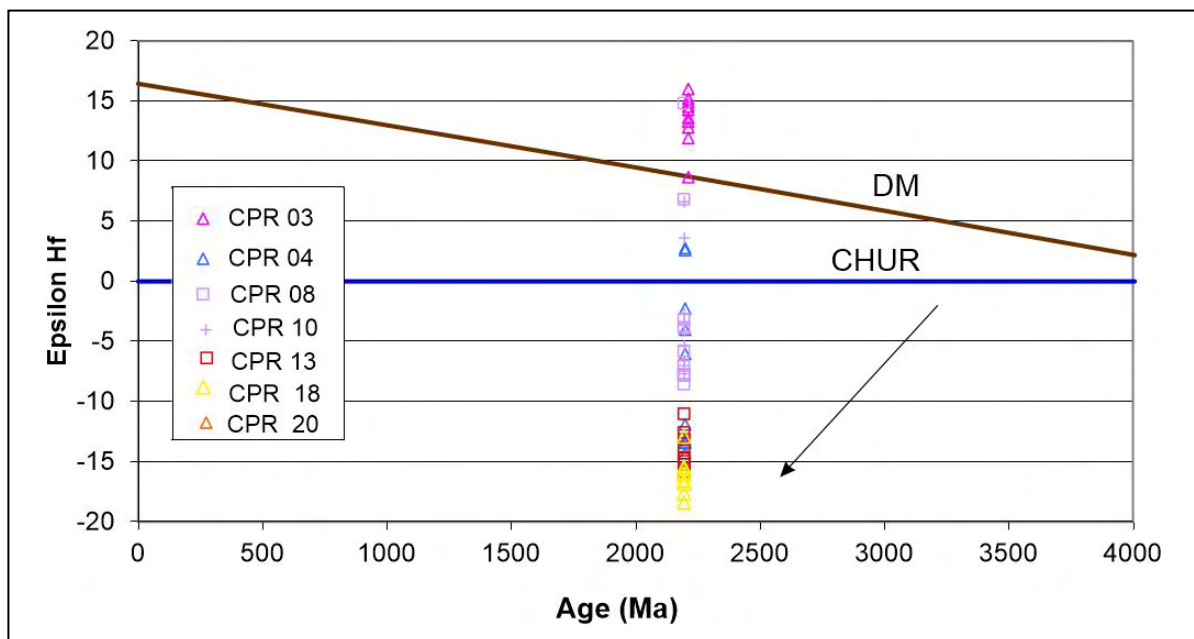


FIGURE 14. Hf isotopic evolution diagram for the studied samples. It is observed the variation of Hf isotope values in the Paleoproterozoic zircon grains, indicating a mantle source with an important crustal contribution for the magma formation of the Caparaó Complex rocks. The arrow indicates the isotopic evolution of Hf for the ratio $^{176}\text{Lu} / ^{177}\text{Hf} = 0.015$ (Belousova et al. 2010).

2006). In contrast, zircon grains grown in anhydrous metamorphic environments present Th/U ratios preserving the original protolith compositions. While partial loss of radiogenic Pb is frequently observed, examples of almost complete loss of this element are interpreted as chemical leaching during extreme mylonitization process (Wayne and Sinha 1988).

Thus, the hydrothermal fluid is a facilitating medium for the isotopic homogenization and conditions for zircon growth (Rowley et al. 1997) and the distribution of U and Th in zircon allows the identification of fluid availability (fluid inclusions, structural hydroxyl, and crystalline water) within the crystals of high-grade metamorphic rocks (Chen et al. 2010).

Since fluid availability controls zircon growth during high-grade metamorphism, the concentrations of U and Th in metamorphic domains of different zircon grains or among distinct grains reflect the activity of the fluid involved responsible for the metamorphism.

The CL images of the zircon grains studied in this work indicate that most grains nuclei are of igneous origin, but some of them underwent different degrees of metamorphic modification. Such domains are present in zircon grains as edges where no oscillatory zoning typical of magmatic domains is observed. Figure 15 presents the relationship between the $^{207}\text{Pb}/^{206}\text{Pb}$ apparent age and the Th/U ratios for the studied samples. It shows that the inherited nuclei with the isotopic signature of the protolith and the edges that underwent metamorphic re-homogenization show equivalent values of U and Th. The values of U and Th in these domains suggest the non-interference of fluids during the metamorphic process, since this should have been an anhydrous event, as corroborated by petrographic analyzes and the mineralogy composed of orthopyroxene + K-feldspar + garnet and amphibole associated only to mylonitic zones.

7. Conclusion and final remarks

The analytical techniques U-Pb and Lu-Hf were applied to understand the crustal evolution of the Caparaó Complex rocks. The Lu-Hf isotopic analyses contributed to the understanding of the magmatism origin involved in the rocks formation of the Caparaó Complex (ϵ_{Hf} petrogenetic parameter) and to the understanding of the period of

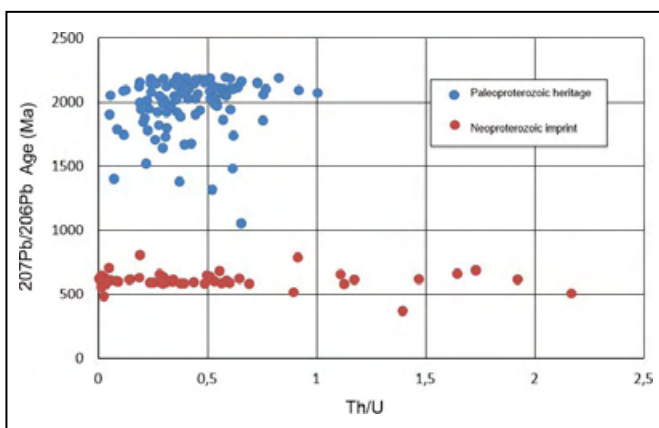


FIGURE 15. Relationship between the $^{207}\text{Pb}/^{206}\text{Pb}$ apparent age and the Th/U ratios for the studied samples. The results show little variation in the isotopic signatures for the inherited domains and newly formed domains in the zircon grains.

mantle extraction (T_{DM} 'crustal age') of such magmatism. The present investigation provided robust Lu-Hf isotopic data (model ages and ϵ_{Hf} parameter) poorly observed in the literature. It also provided intervals of U-Pb ages for the Caparaó Complex, between 2209 and 2060 Ma, like those reported in the literature for the same rocks of 2195 ± 15 Ma. The results reported herein allow to correlate the Caparaó Complex to the Juiz de Fora Complex, with granulitic rocks with age of 2199 ± 17 Ma and important rework associated with the Brasiliano Orogeny.

It is noteworthy that the data obtained in this work corroborate the proposals of Horn (2006) and Novo et al. (2011) that the Caparaó Complex configures a tectonic Paleoproterozoic fragment reworked in Brasiliano Orogeny, during collisional events. With the obtained geochronological data, it is possible to propose a geological evolution of the Caparaó Complex in 3 evolutionary stages:

First stage: 2209 – 2060 Ma (Magmatic Arc)

The Paleoproterozoic ages obtained in the Caparaó Complex represent a period of crustal accretion with juvenile sources (mantle-derived) in a magmatic arc environment (Figure 16 A) with signature of intraoceanic environments (medium-high-K calcium-alkaline rocks similar to that reported by Novo et al. 2011). This event has a strong correlation with the ages obtained in Mineiro Belt (Ávila et al. 2010) and in Juiz de Fora Terrane rocks (Heilbron et al. 2004). This event may be responsible for the amalgamation of São Francisco craton in a global paleocontinent. Supercontinent collage during the Paleoproterozoic times is indicated by paleomagnetic data during the Columbia amalgamation which includes Laurentia (Superior and Wyoming cratons), Baltica (Karelia craton), Australia, and Kalahari and Kaapvaal cratons at about 2.45 Ga (Rogers 1996; D'Agrella Filho et al. 2020). A second continental mass is proposed at that period of time (Pesonen et al. 2003). The break-up of Columbia supercontinent resulted in formation of platform sedimentary basins (Figure 16B).

Second stage: Rio Negro Magmatic Arc

The set of ages from 640-580 Ma represents the oldest stage of rework generated during the Brasiliano event (Figure 16 C). It can be correlated with the end of the implantation of the Rio Negro magmatic arc (Pedrosa-Soares 2001; Tupinambá et al. 1996, 2012). In this way, at this time interval the Rio Negro magmatic arc developed in the Ribeira Orogeny (790-620 Ma). Such a segment would have developed over a prolonged period of subduction and collision between the blocks of western Gondwana in SE Brazil and West Africa. This event is also discussed by Pedrosa Soares (2001) in the Araçuaí belt as pre-collisional or pre-orogenic stage that occurred at about 630 to 585 Ma.

Third Stage: 633 - 584 Ma (Brasiliano collisional stage)

This stage represents the peak of the metamorphic deformation of the Caparaó Complex rocks and is correlated with the acting of the thrusting zones that controlled the regional structure (Figure 16 D). As several

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