

## Detrital zircon U–Pb geochronology constrains the age of Brazilian Neogene deposits from Western Amazonia

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### ABSTRACT

The fossiliferous beds of the Solimões Formation, western Brazilian Amazon have yielded several vertebrate fossils that are key to understand the evolution of Neotropical biotas. Although this sedimentary unit has been studied for more than two centuries, no absolute dates are available so far, preventing more precise bio/chronostratigraphic interpretations and broader comprehension of the biotic/geological events that affected this northern portion of South America during the Neogene. Here, we present the first Neogene radioisotopic dates for the Brazilian Amazon, via U-Pb dating of detrital zircon from two classical sites. Both samples have small proportions of relatively young zircon grains, the dates from which are the maximum ages of deposition. LA-ICPMS analysis of two grains from the Niterói locality yielded a weighted mean of  $8.5 \pm 0.5$  Ma and CA-TIMS analysis of two grains from the Talismã locality yielded a weighted mean of  $10.89 \pm 0.13$  Ma. These maximum deposition ages are in the Tortonian stage, late Miocene, confirming the age previously inferred based on biochronological data.

### 1. Introduction

The Miocene epoch (23.03–5.333 Ma; Cohen et al., 2013 updated) was a critical time interval for the diversification of the Neotropical biota and the origin of several vertebrate groups in western Amazon (Antoine et al., 2016; Tejada-Lara et al., 2015). Significant events affected the Neotropics in that epoch, including the first steps of the Great American Biotic Interchange (GABI) and the origin of the proto-Amazon river. The former led to important changes in the American ecosystems, with a higher impact in the mammalian faunas (Carrillo et al., 2015; Cione et al., 2015; Goin et al., 2012; O'Dea et al., 2016; Woodburne, 2010; Woodburne et al., 2006). It is key to precisely date the strata documenting the first steps of this event, as to understand the impact of the North American immigrants in the South American faunas.

Miocene biotas are known from several sites across South America, those from the northern portion of the continent having been found in

Colombia (Kay et al., 1997), Venezuela (Sánchez-Villagra and Aguilera, 2006), Peru (Antoine et al., 2016, 2013; Campbell et al., 2006, 2001), and Brazil (Negri et al., 2010b). Most of those strata record continental environments, but short-lived marine incursions are also reported (Jaramillo et al., 2017 and references therein). In Brazil, the Solimões Formation, in western Amazon, has yielded vertebrate fossils since the late 1800s, contributing to understand the origin of the modern Neotropical biota (Alvarenga and Guilherme, 2003; Cozzuol, 2006; Frailey, 1986; Hsiou and Albino, 2009; Kerber et al., 2016, 2017, 2018; Negri et al., 2010b; Rancy, 1985; Ribeiro et al., 2013a; Riff et al., 2010; Rodrigues, 1892; Simpson and Paula Couto, 1981). The upper strata of this sedimentary unit are interpreted as deposited during the late Miocene, based on tetrapod biochronology and palynology (see Cozzuol, 2006; Kerber et al., 2017; Latrubesse et al., 1997, 2010, 2007; Negri et al., 2010a; Ribeiro et al., 2013b). The presence of older strata also has been inferred based on the biochronological data provided by

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some taxa found in the Juruá river (see Ribeiro et al., 2013b; Kerber et al., 2017), but no absolute ages had so far been provided for the Solimões Formation.

Although the Solimões Formation is one of the best-sampled Neogene fossil-bearing stratigraphic units of northern South America, assumptions on the age of deposition were only based on indirect methods. The absence of absolute ages hampers more refined interpretations on the paleoenvironments and paleoecology of the faunistic associations found there and does not allow for answering some major key questions, as if these beds were deposited after, during, or before the GABI, or if the proto-Amazon river was already formed during their deposition. To help to answer these and other questions, we present the first detrital zircon U-Pb geochronology for the Solimões Formation (Brazil). The samples were collected in two of the best-sampled paleontological sites of the region, the Niterói (Acre River) and Talismã (Purus River) localities (Souza-Filho and Guilherme, 2015).

## 2. Material and methods

### 2.1. Localities

The Acre Basin is located in northwestern Brazil, neighboring Peru and Bolivia, and preserves a Paleozoic–Pliocene stratigraphic sequence (Cunha, 2007). The Solimões Formation, exposed in the southwestern Brazilian Amazon (States of Acre and Amazonas), encompasses the entire Cenozoic sedimentary record of the basin.

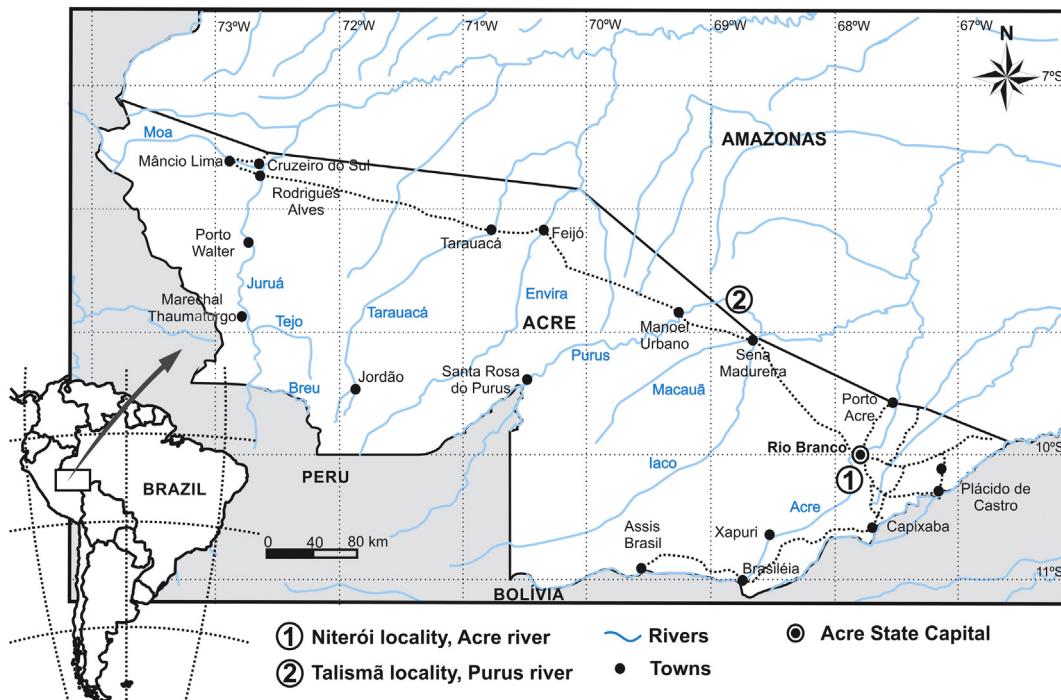
The Niterói locality is located on the right embankments of the Acre River, Senador Guiomar municipality (UTM 19L 629983 E/8879539 S, datum WGS84, Fig. 1). Discovered in 1987, it is one of the most important Neogene fossiliferous sites of the Brazilian Amazon (Bocquentin-Villanueva et al., 1990; Gayet et al., 2003; Latrubesse et al., 1997, 2010; Mones and Toledo, 1989; Negri and Ferigolo, 2004, 1999), with a rich and diverse paleofauna, including crocodylians (Gasparini, 1985; Souza-Filho and Bocquentin-Villanueva, 1991), rodents (Kerber et al., 2017, 2018), xenarthrans (Bocquentin and

Guilherme, 1999; Frailey, 1986), characid fishes (Monsch, 1998), turtles (Bocquentin et al., 2001), and birds (Alvarenga and Guilherme, 2003). Deposits in the site are characterized by an 11 m thick massive and conglomeratic clay, with silt layers with gypsum veins, and two fossiliferous levels (Latrubesse et al., 1997). More recently, the outcrop has been covered by dense vegetation, allowing the visualization of only two clay layers and one fossiliferous level (Fig. 2). These are typical of the floodplain-lacustrine, low energy facies found in most Neogene outcrops of the Brazilian Amazon (Cozzuol, 2006; Latrubesse et al., 2010).

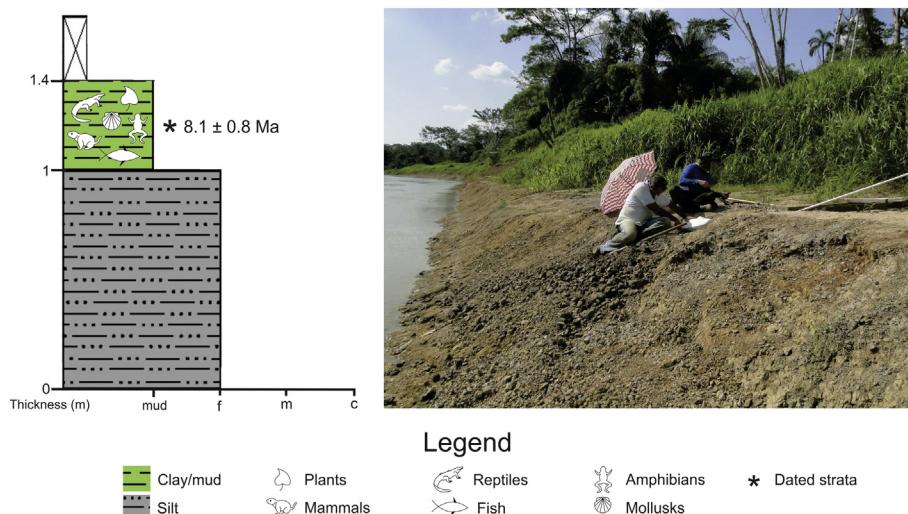
The Talismã locality is located on the right embankments upstream of the Purus River, State of Amazonas, between the Iaco River mouth and the town of Manuel Urbano (UTM 19L 510475 E/9029741 S, datum WGS84, Fig. 3) (Bergqvist et al., 1998; Bocquentin-Villanueva and Negri, 1993; Santos et al., 1993). It was discovered in 1988 and the recovered fossils include crocodylians (Fortier et al., 2014), squamates (Hsiou et al., 2009, 2010; Hsiou and Albino, 2009), rodents (Bergqvist et al., 1998; Kerber et al., 2016, 2018), xenarthrans (Negri, 2004), litopterns (Bergqvist et al., 1998), primates (Bergqvist et al., 1998), and birds (Alvarenga and Guilherme, 2003). The section is dominated by fine sediments (clays and silts) with no lamination or other recognizable structures. Gypsum and calcite veins are present, possibly diagenetic origin (Cozzuol, 2006). The entire exposed section is 5.30 m thick, yielding three fossiliferous layers (Fig. 3).

### 2.2. U-Pb geochronology

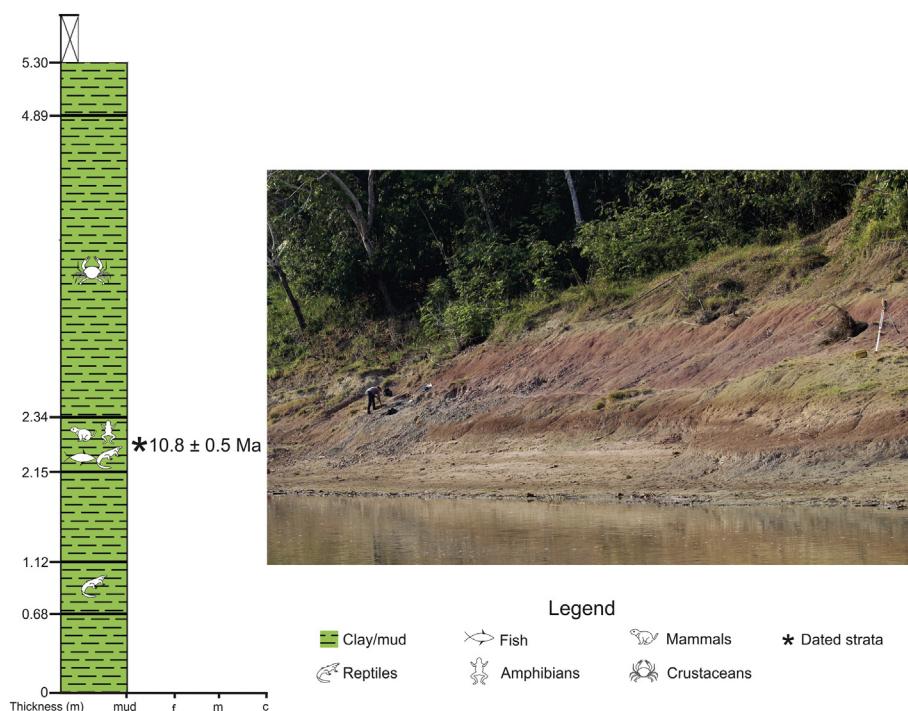
Zircon grains were separated from the mudstone rocks using standard techniques and annealed at 900 °C for 60 h in a muffle furnace. They were mounted in epoxy and polished until their centers were exposed. Cathodoluminescence (CL) images were obtained with a JEOL JSM-1300 Scanning Electron Microscope and Gatan MiniCL. Zircon was analyzed by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) using a ThermoElectron X-Series II quadrupole ICPMS and New Wave Research UP-213 Nd:YAG UV (213 nm) laser



**Fig. 1.** Location of the Niterói and Talismã localities (Solimões Formation), western Brazilian Amazon (Acre and Amazonas states). Full line = political boundaries; dotted lines = main roads; blue lines: main rivers of the Amazon region. Location of the Niterói and Talismã localities (Solimões Formation), western Brazilian Amazon (Acre and Amazonas states). Full line = political boundaries; dotted lines = main roads; blue lines: main rivers of the Amazon region. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 2.** Stratigraphic profile of the Solimões Formation at Niterói locality, Acre River. \* maximum depositional age from the weighted mean of LA-ICP-MS dates from two zircon grains.



**Fig. 3.** Stratigraphic profile of the Solimões Formation at Talismā locality, Purus River. \* maximum depositional age from two zircon grains analyzed by CA-TIMS.

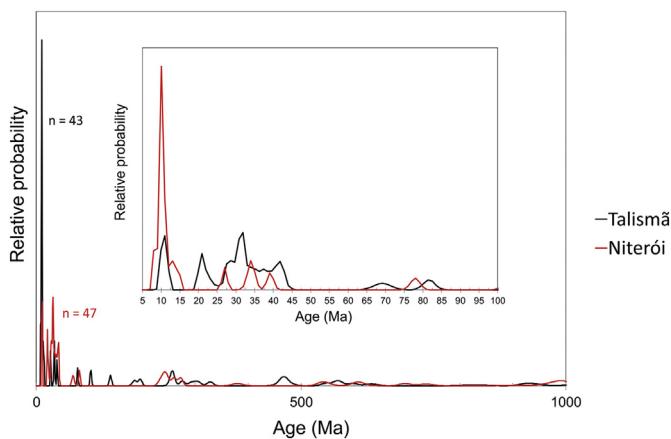
ablation system. The two youngest grains from each sample were then analyzed by chemical abrasion isotope dilution thermal ionization mass spectrometry (CA-TIMS) using a GV Isoprobe-T multicollector mass spectrometer equipped with an ion-counting Daly detector. Details of the analytical procedures, performed at the Isotope Geology Laboratory (IGL) at Boise State University, are given in Supplemental materials (Supplemental data and Tables S1 and S2).

Weighted mean  $^{206}\text{Pb}/^{238}\text{U}$  dates were calculated from equivalent dates (probability of fit  $> 0.05$ ) using Isoplot 3.0 (Ludwig, 2003). Errors on the weighted mean CA-TIMS dates are given as  $\pm x/y/z$ , where  $x$  is the internal error based on analytical uncertainties only, including counting statistics, subtraction of tracer solution, and blank and initial common Pb subtraction,  $y$  includes the tracer calibration uncertainty propagated in quadrature, and  $z$  includes the  $^{238}\text{U}$  decay constant uncertainty propagated in quadrature. Internal errors should be

considered when comparing our dates with  $^{206}\text{Pb}/^{238}\text{U}$  dates from other laboratories that used the same EARTHTIME tracer solution or a tracer solution that was cross-calibrated using EARTHTIME gravimetric standards. Errors including the uncertainty in the tracer calibration should be considered when comparing our dates with those derived from other geochronological methods using the U-Pb decay scheme (e.g., laser ablation ICPMS). Errors including uncertainties in the tracer calibration and  $^{238}\text{U}$  decay constant (Jaffey et al., 1971) should be considered when comparing our dates with those derived from other decay schemes (e.g.,  $^{40}\text{Ar}/^{39}\text{Ar}$ ,  $^{187}\text{Re}-^{187}\text{Os}$ ). Errors for weighted mean dates and dates from individual grains are given at  $2\sigma$ .

### 2.3. Abbreviations

SALMA, South American Land Mammal Age; U-Pb,



**Fig. 4.** Normalized relative probability plot of LA-ICP-MS zircon dates from Talismã and Niterói fossiliferous localities, with inset plot showing dates of  $\leq 100$  Ma grains. Ten  $> 1000$  Ma dates are not shown.

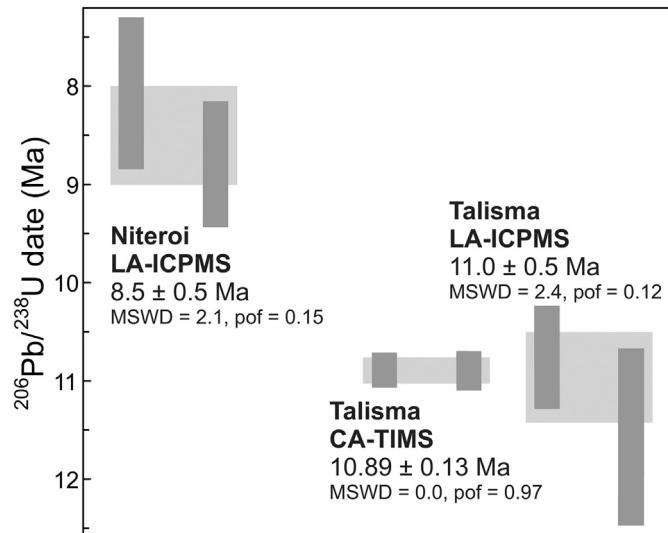
uranium-thorium;  $^{40}\text{Ar}/^{39}\text{Ar}$ , argon-argon;  $^{87}\text{Sr}/^{86}\text{Sr}$ , strontium-strontium.

### 3. Results

#### 3.1. U-Pb geochronology

LA-ICP-MS ages from 47 zircon grains from Niterói are between  $1804 \pm 72$  and  $8.1 \pm 0.8$  Ma. The youngest thirteen dates range between  $14.4 \pm 0.9$  and  $8.1 \pm 0.8$  Ma (Figs. 4 and 5). The youngest two dates are equivalent at  $8.8 \pm 0.6$  and  $8.1 \pm 0.8$  Ma and yield a weighted mean date of  $8.5 \pm 0.5$  Ma (MSWD = 2.1, probability of fit = 0.15). This is interpreted as the maximum depositional age. CA-TIMS was attempted on these two grains, but due to significant loss of volume during chemical abrasion, the fragments had too little radiogenic Pb to yield useful dates.

LA-ICP-MS ages from 43 zircon grains from Talismã range between  $2135 \pm 38$  and  $10.8 \pm 0.5$  Ma (Figs. 4 and 5). The youngest two dates are equivalent at  $11.6 \pm 0.9$  and  $10.8 \pm 0.5$  Ma and yield a weighted mean date of  $11.0 \pm 0.5$  Ma (MSWD = 2.4, probability of fit = 0.12). CA-TIMS dates from these two grains are equivalent with a weighted



**Fig. 5.** Plot of the youngest  $206\text{Pb}/238\text{U}$  dates obtained by CA-TIMS and LA-ICPMS. Plotted with Isoplot 3.0 (Ludwig, 2003). Error bars are at 2 sigma. Weighted mean dates are shown and represented by the grey boxes behind the error bars.

mean of  $10.89 \pm 0.13/0.13/0.13$  Ma (MSWD = 0.0, probability of fit = 0.97). This is interpreted as the maximum depositional age.

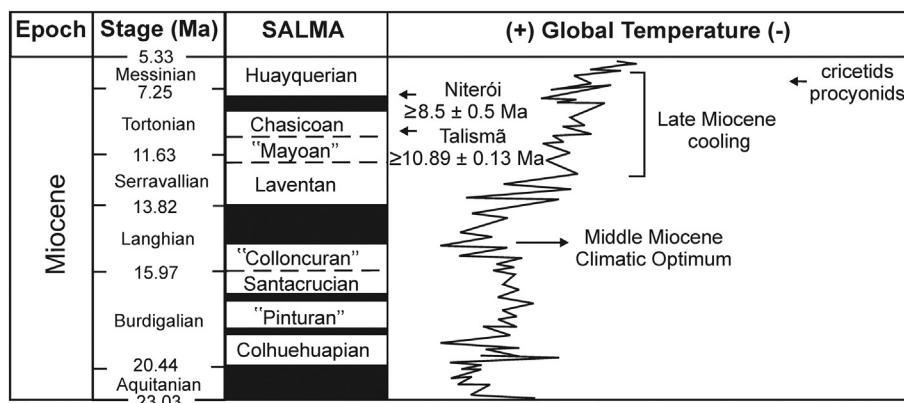
### 4. Discussion

#### 4.1. Vertebrate fossil content of Talismã and Niterói localities and biochronology

The fossil vertebrates of the Acre Basin have been studied for over 120 years and they document a rich Neogene taxonomic diversity. Discovered in the 1980's by UFAC (Universidade Federal do Acre) team and North American researchers (Natural History Museum of Los Angeles County), the Talismã and Niterói sites are two of the best sampled fossiliferous localities of the Solimões Formation (Cozzuol, 2006; Latrubbles et al., 2010; Souza-Filho and Guilherme, 2015). These have yielded dozens of vertebrate specimens, but their fossil content is not the same, especially considering the mammal fauna (Negri, 2004; Cozzuol, 2006; Kerber et al., 2017).

Among the many taxa recorded in these localities, xenarthrans and rodents are those that provide a better basis for comparison between the fossil content of both sites and other South American faunas of similar age. Based on their general "evolutionary degree", Negri (2004) suggested that the ground sloth fauna of the Talismã locality, represented by *Octodontobradys puruensis* Santos, Rancy, and Ferigolo, 1993 (Orophodontidae), cf. *Hapalops* Ameghino, 1887 (Nothrotheriinae), and cf. *Planops* Ameghino, 1887 (Megatheriidae), is older than its supposed late Miocene age or represents a relictual association that survived for a longer time, because orophodontids are usually found in late Oligocene Deseadan beds, and *Hapalops* and *Planops* are present in the Santacrucian SALMA (early Miocene) of Argentina (Negri et al., 2010b). This interpretation led Negri (2004) to infer at least a Laventan age for the fauna found in Talismã. On the other hand, Negri (2004) observed that the ground sloths found in Niterói, including *Urumacotherium campbelli* (Frailey, 1986) and *Pseudoprepotherium venezuelanum* Hoffstetter, 1961, have affinities with Huayquerian SALMA (late Miocene) forms from Urumaco, Venezuela (see Bocquentin and Guilherme, 1999; Negri, 2004; Negri et al., 2010a; Negri and Ferigolo, 2004). However, more recently in a comprehensive work of well-constrained localities of Contamana region, Antoine et al. (2016) present records of Miocene orophodontids from deposits of Pebas Fm. in Contamana region (CTA-63 - early Miocene, Colhuehuapian SALMA; CTA-57 - earliest late Miocene, Mayoan SALMA), and ground sloths remains closed allied to *Pseudoprepotherium* were also recorded in early Miocene deposits of Contamana region (CTA-63 - Colhuehuapian SALMA, Antoine et al., 2016). Concerning rodents, Kerber et al. (2017) recently reviewed the fossil record of the group in western Brazilian Amazon. The authors emphasize that the fossil content of both deposits differ. Among the differences, Talismã yielded *Neoepiblema horridula* and *Drytomomys* sp., whereas Niterói houses *Neoepiblema acreensis*, with more derived traits (Bocquentin-Villanueva et al., 1990; Kerber et al. in press). *Drytomomys* is particularly interesting because it appears in middle Miocene beds (Laventan) of northern South America like the Fitzcarrald arch region and deposits of the Pebas Fm. in the Peruvian Amazon (Fields, 1957; Walton, 1997; Tejada-Lara et al., 2015; Antoine et al., 2016, 2017; Kerber et al., 2017).

Considering the maximum age of deposition here reported, both localities were deposited, at most, during the Tortonian (Chasicóan/Huayquerian SALMAS) (Fig. 6), contrary to the previous interpretations that Talismã locality could expose middle Miocene levels (Laventan SALMA (e.g. Negri, 2004)). Nevertheless, Talismã might be  $\sim 1.5$ – $2$  million years older than Niterói, when considering similar temporal gaps between zircon crystallization in the Andes and their final deposition in the concerned Amazonian localities. Although we are working with detrital zircon and, consequently, with estimates of maximum depositional ages, this interpretation is corroborated by faunal dissimilarities between both localities. The ages presented here



**Fig. 6.** Maximum depositional U-Pb ages from Solimões Formation (Niterói and Talismā) in the context of the Neogene SALMAS, climatic changes, and the arrival of the first Holarctic mammals immigrants. Modified from Goin et al. (2016: Fig. 4.6).

are similar to the  $^{40}\text{Ar}/^{39}\text{Ar}$  ( $9.01 \pm 0.28$  Ma) dates reported by Campbell et al. (2001) for the volcanic Cocama Ash (upper Purus river, Madre de Dios Formation), Peru, a site located at the Peruvian portion of the same river where Talismā is placed. The variation in the ages within the Tortonian corroborates previous interpretations (Cozzuol, 2006; Latrubesse et al., 2007, 2010) about possible diachronisms among the fossil assemblages of Acre basin during the late Miocene. However, further assessment of diachronism within Miocene deposits of the Solimões Fm. requires additional dates from other localities and different lithologies.

#### 4.2. Detrital zircon ages and the comprehension of the main biotic/geological events during the late Miocene of western Amazonia

It is noteworthy that no confident fossils of Holarctic immigrants have been found in the deposits of the Solimões Formation (Cozzuol, 2006; Latrubesse et al., 2010). Some putative records of north immigrants have been recorded in the upper Miocene of Brazil and Peru (Campbell et al., 2010, 2001; Frailey and Campbell, 2012; Prothero et al., 2014), but their taxonomic identity, age, and stratigraphic provenance are still debated (Alberdi et al., 2004; Antoine et al., 2016; Dutra et al., 2017).

Recent reviews on the paleontological data of mammals in the GABI agree that this event was a gradual process that began during the late Miocene (~10-7 Ma) (Carrillo et al., 2015; O'Dea et al., 2016) by organisms that could disperse from North America to South America through rafting. After the total closure of the isthmus, the faunal interchange was clearly intensified (~2.8 Ma) (O'Dea et al., 2016; but see Bacon et al., 2015; Montes et al., 2015). Besides the still doubtful records from Amazonia above mentioned, procyonids and cricetids are the first mammals with Holarctic origin and confident fossil record (Cione et al., 2007; Goin et al., 2012; Forasiepi et al., 2014; Prevosti et al., 2013; Reguero and Candela, 2011; Carrillo et al., 2015; O'Dea et al., 2016) that dispersed to South America by rafting (O'Dea et al., 2016). The oldest South American procyonids are from the late Miocene of northwest and northeast Argentina (~7 Ma, Reguero and Candela, 2011), while in the tropics they appear only in the Pliocene fossil record from Venezuela (Forasiepi et al., 2014). In the Ituzaingó Fm., Entre Ríos, Argentina, which is one of the Miocene localities with fossil content sharing several taxa with the Solimões Formation (Cozzuol, 2006), procyonids are represented by *Cyonasua argentina* Ameghino, 1885 (see Soibelzon and Bond, 2013). This sedimentary unit assigned to the Tortonian (Cione et al., 2000), has no associated absolute ages. However, this formation overlies the Paraná Formation dated by  $^{87}\text{Sr}/^{86}\text{Sr}$  revealing a Tortonian age of 9.47 Ma (Pérez, 2013), which suggested that the Ituzaingó Fm. is at least younger than this. The oldest cricetids are also from Argentina. Although there is a debate about the

age of some Neogene records (late Miocene - Verzi and Montalvo, 2008; or Pliocene - Prevosti and Pardiñas, 2009), Nasif et al. (2009) reported a fragmentary material assigned to Cricetidae indet. from the upper Miocene levels of the Andalhualha Fm., Catamarca Province, collected near to ash levels dated in 7.14 Ma (Messinian). Despite the fragmentary nature of the specimen, it is still being the oldest evidence of this clade in South America. Non-doubtful records of cricetids are derived from levels aged ~5 Ma (Pardiñas and Tonni, 1998; Prevosti and Pardiñas, 2009). Therefore, if we consider that the oldest fossils of Holarctic mammals are in the Tortonian/Messinian, the fauna found in the Solimões Fm. (at least for the localities dated here, or others with similar fossil record) is slightly older or within the time interval that documents the appearance of these organisms in South America. Hence, we do not rule out the possibility that these groups that have reached South America by oceanic dispersal may be found in the Tortonian of Amazonia.

During the late Miocene geological events such as the Andes uplift and the origin of the proto-Amazon river (Aguilera et al., 2017; Hoorn et al., 2017), led to profound changes in the ancient biota of the Amazon region (Hoorn et al., 2010; Latrubesse et al., 2010; Silva-Caminha et al., 2010). The oldest record of Andes-derived sediments in the Amazon delta is 9.4 Ma old (Hoorn et al., 2017), which is in line with the proposed drainage changes of the Amazon Basin during the late Neogene (Hoorn et al., 2010). In the last 10 Ma, due to the global sea level drop, the cooling temperature trends, and the Andean uplift, the drainage in western Amazon changed from a lake-dominated (Pebas system) to a fluvial-tidal-dominated system (Acre system) (Antoine et al., 2007, 2016, 2017; Hoorn et al., 2010; Wesselingh et al., 2002). A still debated question is for how long the lake-dominated ecosystems persisted in the northernmost Neotropics (Salas-Gismondi et al., 2015; Wesselingh et al., 2006). Salas-Gismondi et al. (2015), in a comprehensive analysis of crocodylian diversity from the middle and late Miocene of northeastern Peru, correlate the hyperdiverse crocodylian community with wetland environments. The same environmental interpretation was reached by Gross et al. (2013) based on the isotopic analyses of ostracods shells.

Both Niterói and Talismā yield crocodylian taxa whose feeding ecology is also related to lentic environments (e.g. *Mourasuchus* sp. and gavialoids) and large flooded areas (e.g. *Purussaurus brasiliensis*) (see Cidade et al., 2017 for more details). Based on Niterói maximum age of deposition, we can conclude that even with the onset of proto-Amazonas (9.4 Ma, Hoorn et al., 2017), there were up to 8 million years ago flooded areas with sufficient extension for the maintenance of this diverse crocodylian fauna.

Thereby, the fossil assemblages of the Solimões Formation are of paramount importance to understand the Amazonian faunal and environmental changes at the Miocene/Pliocene transition, which are

closely related to modern Amazon rainforest biodiversity (Haffer, 2008; Hoorn et al., 2010). Nevertheless, without rigorous excavation protocols, including provenance information (Bissaro-Júnior et al., 2018), correlating fossil-bearing rock strata with absolute dates is nearly impossible.

## 5. Conclusions

For the first time, two of the most important fossil-bearing localities of the Solimões Formation are constrained in age. U-Pb dates from detrital zircon grains confirm that these strata were deposited at most during the Tortonian, late Miocene. Based on both faunal dissimilarities and maximum depositional age differences between the two localities, we suggest that Talismã is older than Niterói, however we stress the need for further zircon dating to test this hypothesis, as well as dating efforts from other localities and different lithologies to test further depositional diachronisms in the Solimões Fm.

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.palaeo.2018.11.032>.

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