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Refining the chronology of Middle/Late Pleistocene fossil assemblages in the Argentine Pampas

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ABSTRACT

Middle and Late Pleistocene large and mega-mammal records of the Argentine Pampas, belonging to the Bonaerian and Lujanian Stages/Ages, have not yet been adequately dated by standard numerical radiometric methods. Reference timescales for Argentina and other South American fossil sites are mainly based on the mammalian faunal content (biostratigraphic units or biozones). We have recently dated three Middle and Late Pleistocene faunal sites located in the Buenos Aires Province using Electron Spin Resonance (ESR), U-series, Optically Stimulated Luminescence (OSL) and radiocarbon dating. The geographically distant sites (Salto de Piedra, Campo Spósito and Cascada de Paleolama) have yielded fossil assemblages representative of the Lujanian and Bonaerian Stages/Ages. Salto de Piedra, systematically excavated, records one of the most complete fossil sequences and our results indicate that it deposited between at least 127.4 ka (minimum age; Late Pleistocene, MIS 5) and 4.8–4.3 cal ka BP (Middle Holocene, MIS 1). Campo Spósito lower level (corresponding to 'El Tala' unit) was dated to >188 ka via ESR/U-series dating of teeth. Finally, Cascada de Paleolama Unit D, towards the base of the sequence, produced an age of 143 ± 20 ka (Late Pleistocene, MIS 5/6). The new dating results provide a crucial additional age control for the Bonaerian and Lujanian Stages/Ages, and allowed us to better identify and calibrate the limits of *Equus neogeus, Megatherium americanum* biozones, as well as to better constrain other index taxa that are used to construct local biostratigraphical frameworks. Our results indicate the need for a revision of the biozone schemes established for the Quaternary of Pampean region.

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1. Introduction

The Argentine Pampean region is a wide region of central-east Argentina, covering ~370,000 km^2. This biogeographical region, bear specific fauna and flora characteristic of the Pampean plains and is divided into two subregions, the Humid Pampa towards the southeast, and Dry Pampa in the West (Morrone, 2001; Prieto, 2000). The Pleistocene and Holocene sedimentary deposits in the Pampean region, particularly in the Buenos Aires Province (Fig. 1), have traditionally been assigned to different locations that provided information on the evolution of the paleontological and geological records. Between 1869 and 1878, the naturalist Florentino Ameghino studied the Quaternary outcrops of the Luján River and adjacent areas to gather evidence about the coexistence of the first human groups with the extinct megafauna. Almost simultaneously with Ameghinino's first scheme, Doering (1882) grouped the surface sedimentary deposits into 7 formations and 14 horizons. Ameghino (1881, 1884, 1889) proposed a hierarchical sequence of alluvial units defined according to their paleontological content and lithology, which were divided into a succession of stages or horizons.

Considering the difficulties in establishing limits based on lithology, Ameghino (1881) proposed a subdivision of the Pampean Formation into three units characterized by their fossils content, from bottom to top: "Pampeano Inferior", "Pampeano Superior" and "Pampeano Lacustre". Later, Ameghino (1884–1889) named the sedimentary deposits according to Doering (1882) as "formación pampeana", with the following "stages": "Ensenadense" or Ensenadan (="Pampeano Inferior"), "Belgranense" or Belgranian (="Pampeano Medio"), "Bonaerense" or Bonaerian (="Pampeano Superior"), "Lujanense" or Lujanian (="Pampeano Lacustre"), "Platense" or Platan (="Postpampeano Lacustre"), and "Aymará" or Aymaran (see Table 1).

Later, Fidalgo et al. (1973) established a lithostratigraphic scheme for the Late Pleistocene based on the sequence he described in the Salado River (that flows into Samborombon bay, Buenos Aires Province). These authors had difficulties in distinguishing the Lujanian and Platan horizons of Ameghino and renamed them as the Guerrero Member (with a lower brown portion and an upper green portion), and the Río Salado Member of the Luján Formation. More recently, Dillon and Rabassa (1985) renamed the brown lower section as La Chumbiada Member, restricting the Guerrero Member to the upper green section (Table 1).

The Cenozoic vertebrate fossil record from the Pampean region has been traditionally divided following two main approaches. On the one hand, Pascual et al. (1965, 1996) proposed biochronological units called "South American Land Mammal Ages" (SALMAs) based on the succession of the evolutionary stage of faunal assemblages and dispersal events (Fig. 2). Later, other authors (e.g., Cione and Tonni, 1995, 1999, 2001, 2005; Cione et al., 2015) proposed a regional chronostratigraphic framework constructed from biostratigraphical studies of the continental sequences from the Pampean region spanning from the Late Miocene to Holocene, making a distinction between geological units (formations) and paleontological content (biozones). This scheme has been developed based on a series of index fossils identified from mammal associations that represent stages/ages (Fig. 2). Recently, modifications to this scheme were proposed by different authors (see Cione et al., 2015; Gasparini et al., 2016; Gasparini et al., 2023, and references therein), but the temporal limits or taxa characterizing the biozones are currently under debate (Fernández-Monescillo et al., 2023; Prevosti et al., 2021; Romano et al., 2023).

Most of the numerical ages available for these sequences were obtained between the 1980s and 2010 using conventional radiocarbon dating, providing highly-needed chronological constraints, but not beyond 35–40 ka. The resulting data set is of uneven quality, and some works do not meet the minimum quality and/or reporting standards in the field. Furthermore, the lack of solid chronological constraints based on numerical dating methods has been worsened by a series of factors, such as the successive changes in the geological time scale and its subdivisions, the diversity of nomenclatures employed by different authors, and the dual classification of deposits based on the content of fossils and

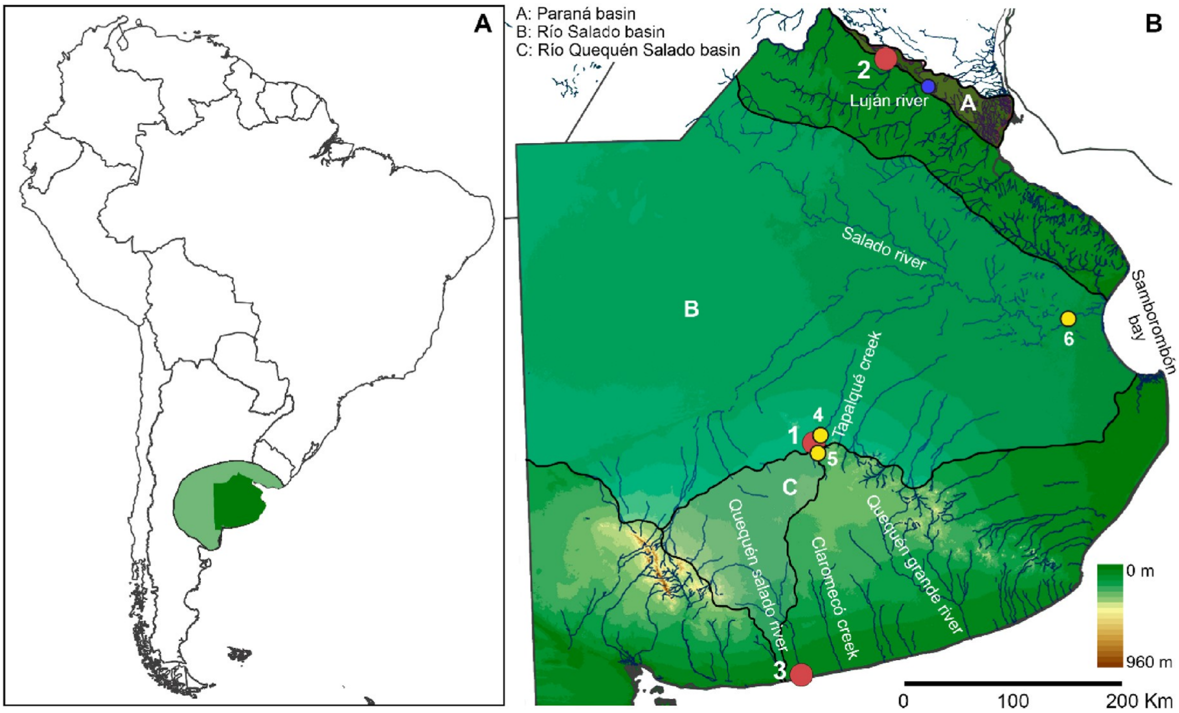

Fig. 1. (A) Map of South America: the Pampean region is shaded in green and in darker green the area shown in Fig. 1B (Buenos Aires Province). (B) Study sites considered in this paper and dated are encircled in red: 1) Salto de Piedra, 2) Campo Spósito (El Tala), 3) Cascada de Paleolama. Other sites mentioned in the text are encircled in yellow: 4) Avenida de los trabajadores, 5) Campo Laborde, 6) Puente de Pascua. Finally, encircled in blue is the locality of the Pampean Formation described by Ameghino (1881).

Table 1
Stratigraphic classifications of the Pampean deposits according to the different authors mentioned in the text.

Ameghino (1880–1881)		Ameghino (1884–1889)	Fidalgo et al. (1973); Fidalgo (1983); Dillon and Rabassa (1985); Tonni et al. (1999); Dangavs and Pierrad (2022); Pommarés et al. (2021)			Estimated Age	[a] Translated Stage names
Formation	Unit	Stage	Formation	Member	Formation		
			Fluvial and lacustrine sediments		Eolian sediments		
Postpampeana	Topsoil	Aymará	Lujan	Río Salado	La Postrera IV	Holocene	Aymaran
	Pospampeano Lacustre	Platense			La Postrera III		Platan
Pampeana	Pampeano Lacustre	Lujanense		Guerrero	La Postrera II	Pleistocene	Lujanian
				Chumbiada	La Postrera I		
	Pampeano Superior	Bonaerense	Pampiano/ Ensenadense	Miembro Superior			Bonaerian
		Belgranense (~Pampeano Medio)					Belgranian
	Pampeano Inferior	Ensenadense		Miembro inferior			Ensenadan

[a] Original Spanish names are translated into English in the last-right column

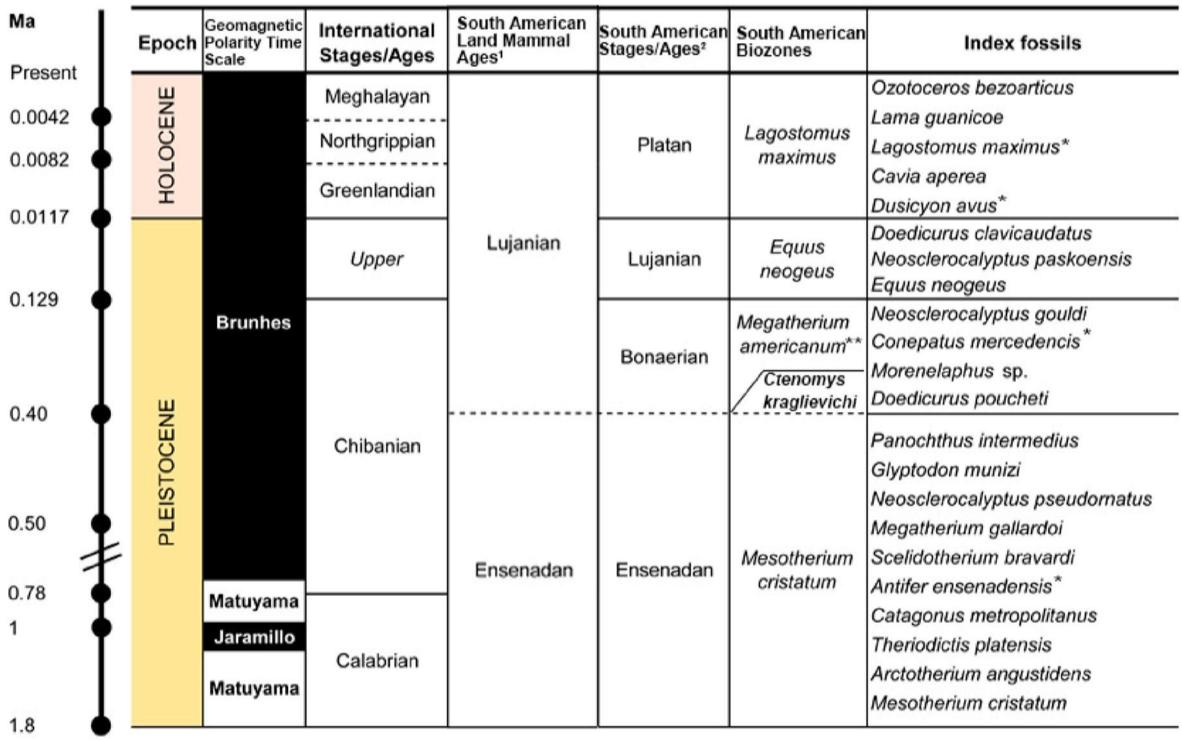

Fig. 2. Chronostratigraphic chart and biozones modified from Cione and Tonni (1995), 1999, 2005, Pascual et al. (1965), Deschamps (2005), Cione et al. (2015), and Gasparini et al. (2023).
Note that one asterisk (*) indicates species that should not be considered exclusive to the biozone as they have a broader biochron than previously considered. Double asterisks (**) refer to *Megatherium americanum*, with fossil remains also recorded in the younger Lujanian Stage/Age (Brandoni et al., 2008).

lithological characteristics (Beilinson et al., 2019).

In this paper, we present new numerical dating results from three Quaternary paleontological sites (Salto de Piedra, Campo Spósito, and Cascada del Paleolama), which are located in different and distant basins of the Pampean region (Fig. 1). Several dating methods (^{14}C AMS, Optically Stimulated Luminescence -OSL-, and Electron Spin Resonance -ESR-, and Uranium series) were employed to date fossils and associated sediments from various stratigraphic units, thus providing key chronological data to improve regional litho- and bio-stratigraphic correlations, and ultimately contributing to a better comprehension of the Pampean biostratigraphy.

1.1. Biozones of the Pampean region, index fossils and first appearance taxa

Fossil taxa that characterize biostratigraphic units (biozones) are known as index taxa, and are either present exclusively in a stratum for a length of time (or biochron), or their first/last appearance concurs in a place and interval characterizing the biozone. Different Association Biozones and Interval Biozones (see CAE, 1992) represent the biostratigraphic basis for the recognition of stages/ages in the Pampean region (see Fig. 2)

Biostratigraphic studies carried out by Cione and Tonni, and collaborators (e.g., Tonni et al., 1992; Cione and Tonni, 2005; Cione et al., 2015; Gasparini et al., 2016; Soilbenzon et al., 2019) have allowed the establishment of a chronostratigraphic-geochronological scheme whose most recent versions include four biozones for the Quaternary of the Pampean region: *Mesotherium cristatum*, *Megatherium americanum/Ctenomys kraglievichi*, *Equus neogeus,* and *Lagostomus maximus*, which are the biostratigraphic basis of the Ensenadan (Early to Middle Pleistocene), Bonaerian (Middle Pleistocene), Lujanian (Late Pleistocene-Early Holocene), and Platan (Early Holocene-sixteenth century) Stages/Ages, respectively.

Recently, Gasparini et al. (2023) revised and updated the biostratigraphy of Quaternary mammals. As a result, some Holarctic immigrants from North-Central America, namely *Epieuryceros*, *Antifer*, *Tapirus*, *Arctotherium*, *Puma*, *Panthera*, *Smilodon*, *Conepatus*, *Cuvieronius* and *Notiomastodon* (*Stegomastodon* in the North), were recorded for the first time within the *Mesotherium cristatum* Biozone. In addition, several taxa are exclusive to this biozone, i.e., *Panochthus intermedius, Glyptodon munizi, Neosclerocalyptus pseudornatus, Neosclerocalyptus ornatus, Eutatus pasquali, Megatherium gallardoi, Scelidotherium bravardi, Catagonus metropolitanus, Theriodictis platensis, Arctotherium angustidens,* and *Mesotherium cristatum.*

In the *Megatherium americanum* Biozone, several biostratigraphically relevant taxa are registered, some exclusive ones (i.e., *Neosclerocalyptus gouldi*, *Doedicurus poucheti*, *Morenelaphus*) and several first records, i.e., *Megatherium americanum*, *Lestodon armatus*, *Mylodon darwini*, *Scelidotherium leptocephalum*, *Glossotherium robustum*, *Glyptodon reticulatus*, *Glyptodon elongatus*, *Panochthus tuberculatus*, *Eutatus seguini*, *Dasypus*, *Macrauchenia patachonica*, *Toxodon platensis*, *Dicotyles tajacu*, *Tayassu pecari*, *Brasiliochoerus stenocephalus*, *Lama gracilis* (Gasparini et al., 2023). The *Ctenomys kraglievichi* Biozone is correlated with the base of the *Megatherium americanum* Biozone. This biozone includes micromammals related to a warm pulse, possibly corresponding to the MIS 11 (ca. 0.4 Ma).

The *Equus neogeus* Biozone is the biostratigraphic base of the Lujanian Stage/Age but is not equivalent to the Lujanian SALMA of Pascual et al. (1965), who included under that name the faunal associations of both the Bonaerian and the Lujanian *sensu stricto*. Gasparini et al. (2023) pointed out that some taxa are exclusively recorded in this biozone, such as *Doedicurus clavicaudatus*, *Neosclerocalyptus paskoensis*, and *Equus neogeus*. The following taxa appear for the first time: *Panochthus greslebini*, *Microcavia australis*, *Dolichotis patagonum*, *Galea leucoblephara* and *Lycalopex gymnocercus*. The earliest records of *Homo sapiens* in the Pampean region occur in this biozone (see Prates et al., 2020), which had a considerable impact on the ecosystems (Prates and Perez, 2021).

In the *Lagostomus maximus* Biozone, only species with currently representatives are registered (both native South American fauna as those that entered during the Great American Biotic Interchange), except for *Dusicyon avus*, which became extinct ca. 400 years ^{14}C BP (Tonni and Politis, 1982; Prevosti et al., 2015). Also, some megamammals species are represented (i.e., *Doedicurus clavicaudatus*), whose most modern records are ca. 8000 years ^{14}C BP (Cione et al., 2009; Politis et al., 2019).

These Pampean biozones share the same problem, i.e., insufficient chronological control for faunas and sediments recorded at well-studied sites. In the last decade or so, some significant efforts have been made to date the fossils and/or the sediments using ESR, combined with Thermoluminescence (TL), OSL, and ^{14}C AMS methods, to re-evaluate these proposed schemes and improve the existing local and regional chronostratigraphic frameworks (Prado et al., 2015; Toledo, 2011; Brunetto et al., 2015; Ferrero et al., 2017; Azevedo et al., 2020). In this paper, we aim to evaluate the reliability of our dating results and those from other publications according to the standards in the field.

1.2. Study sites and regional framework

1.2.1. Salto de Piedra paleontological locality (SPPL)

SPPL is located in the upper basin of the Tapalqué creek (Buenos Aires Province, Argentina), close to Olavarría city (Fig. 1; 36°56′54.6″S, 60°22′19.9″W). This small valley is carved into loessic Plio-Pleistocene sediments, which outcrop discontinuously along the creek banks. The interfluves are covered by a mantle of Late Pleistocene and Holocene aeolian deposits that provide the parent materials for most of today's soils (Prado et al., 2019; Favier-Dubois et al., 2021).

This site is especially important because it is one of the few paleontological sites from the Pampean region that have been systematically excavated and fossils recorded using 3D Total Station coordinates. These recent systematic excavations, combined with stratigraphic and sedimentological studies, were carried out in two specific areas of the stream bank of Tapalqué creek (Salto de Piedra-Section I downstream, and Salto de Piedra-Section II upstream). During the last three years of excavations, an extensive collection of macro-, meso- and micromammals has been recovered. Favier-Dubois et al. (2021) divided the stratigraphic sequence into six depositional units (U) separated by erosive discordances, constituting allounits, and 12 facies that correspond to different sedimentary sub-environments which have different taphonomic conditions concerning the preservation of the fossil record. These allounits, named locally U1 to U6, were identified at the SPPL (Prado et al., 2019) and may be broadly correlated to the upper Pampiano (U1) and Luján (U2 to U5) formations of the geological literature (Fidalgo et al., 1973; Zárate, 2005) based on the discontinuities. The stratigraphy of Salto de Piedra from the base to the top is as follows:

Unit 1 is up to 4.50 m thick and constitutes the creek gully near the waterfall in Section I (excavation Grid 1). The unit is composed of pale brown silty sands. It comprises facies F1 to F3, which are made up of sandy tabular deposits, lenticular gravelly lenses, and diamictes. A well-developed calcrete, up to 40 cm thick, crowns the top of this unit. This calcrete outcrops along the Tapalqué riverbank.

Unit 2 is up to 1.50 m thick and lies in erosive discordance on the calcrete in Section II. It is a pale brown sandy silt tabular deposit (F4), massive to laminated towards the top. A minor overload deformation and thin calcium carbonate lines are visible in these facies (Orgeira et al., 2001).

Unit 3 is up to 2.60 m thick and corresponds to very pale brown sandy gravel to sandy silt deposits represented by facies F5 and F6. The sequence begins with lenticular gravelly lenses of F5 lying in erosive discordance on U2. Above this, sandy tabular deposits developed with horizontal to massive or faint laminated stratification. The top of this unit shows convolute lamination. This deformation affects about 1 m of this deposit and does not appear to have been produced by sediment overload.

Unit 4 is up to 3.40 m thick and comprises brownish yellow to very pale brown gravelly sandy to sandy silt and silty deposits, represented by F7 to F9 facies. This unit has an erosive basal contact. Grain size decreases upward, beginning with sandy lenticular deposits represented by F7 that pass to tabular sandy and silty sediments of F8, culminating with a lenticular silty sand deposit represented by F9. There is an evident change in color between F8 and F9.

Unit 5 is up to 1.50 m thick and comprises a light gray to gray sandy silt to silty clay lenticular deposit represented by F10 and F11. This unit overlays in erosive discordance on U4. Sandy silt laminated to massive deposits predominate.

Unit 6 is up to 0.80 m thick and is a dark grayish-brown sandy silt tabular deposit with a granular structure. This unit was affected by pedogenesis in the form of discrete thin buried A horizons or a melanic cumulic A horizon (Mollisol) that crowns the stratigraphic sequence in Section II.

Several studies were previously carried out to date the fossiliferous levels at SPPL. Bonadonna et al. (1995, 1999), Figini et al. (1995, 1998), and Prado et al. (2015) provided ^{14}C ages (see Table A.1 in the Appendix). These results gave an age of 4.3–6.3 cal ka BP for the uppermost unit of the sequence (U6), while Unit 5 (U5) ranges between 8 and 11.9 cal ka BP, Unit 4 (U4) is about 11.3–16.5 cal ka BP and Unit 3 (U3) is constrained to between 17 and 38 cal ka BP. No age control was available for the lowermost units (U2 and U1), which are beyond the radiocarbon time range.

1.2.2. Campo Spósito fossil site (CSFS/El Tala)

CSFS is located in the southeast area of San Pedro city (33°42′46.3"S, 59°38′2.2"W), North of the Buenos Aires Province (Fig. 1). The site is a quarry on the cliffs that dominate the floodplain of the Paraná, Baradero, and Arrecifes rivers (Toledo et al., 2014). In the nearby town of Baradero, a section was described and divided into eight lithostratigraphic units, assigned to the Bonaerian and Ensenadan formations (Nabel et al., 1993). The lower five units are made of alternating loess and paleosol deposits, while the upper three units are associated with limnic environments.

A 7 m-thick composite stratigraphic column of the Campo Spósito site was published by Toledo et al. (2014) and Toledo (2017), with the following units from the base upwards:

- *Depositional Sequence Set Ensenada.* It consists of pale reddish loessoid deposits with intercalations of greenish palustrine and ephemeral stream sandy facies, that Toledo (2017) tentatively estimated an age >600 ka, although no independent age control is available for this unit.
- *Depositional Sequence El Tala*. It consists of a fining up 1 m thick sequence, constituted by a basal conglomerate of rounded calcrete clasts that fills a fluvial paleochannel. This layer has yielded abundant mammalian fossil remains (some of them in anatomical position), laying in a paleo-fluvial channel (Prado and Alberdi, 2011). Toledo et al. (2014) dated a *Toxodon* tooth and associated calcite deposits from this sequence using ESR and U-series methods, providing a tentative age range of between ~150 and ~200 ka. This chronology should, however, be treated with extreme caution, as the methodology employed, and the results obtained are not reported in accordance with the international standards.
- *Depositional Sequence San Pedro.* It consists of a lower section of interbedded brown silt/sandy silt, with root traces, median to small prismatic structures that grade to the top to massive reddish silts with prismatic structures, filling the paleochannel.
- *Depositional Sequence Belgrano.* It is represented by a thin massive black argillaceous silt layer which is moderately compacted. Toledo et al. (2014) provided a minimum OSL age of >110 ka for this layer (although no methodological details were given) and tentatively correlated this black level to lateral facies of coastal lagoons of the paleo-estuary of the Arrecifes valley corresponding to the MIS 5e maximum flooding event. However, this interpretation needs further analyses and corroboration from the fauna and numerical dating.
- *Depositional Sequence Lujan.* It includes a thick sequence of loess sediments dated at the base by OSL to 50 ka and deposited atop a strong erosional disconformity (see Table A.1; Toledo et al., 2014).

Unfortunately, most of the existing age estimates reported for CSFS do not meet the international reporting standards and should hence be regarded as purely indicative.

1.2.3. Cascada del Paleolama fossil site (CPFS)

The Quequén Salado valleys are excavated into Neogene deposits, discontinuously exposed at various sites along the river. These oldest deposits that outcrop downstream were grouped by Reig (1955) into the informal unit "Irene Formation" and tentatively correlated to the Late Pliocene. Several studies described two faunal assemblages in these deposits: one to the Late Miocene and the other to the Early Pliocene (Beilinson et al., 2017; Pardiñas et al., 2017). Recently, these deposits were dated by $^{40}Ar/^{39}Ar$ to ~5.17 Ma and ~4.33 Ma respectively (Prevosti et al., 2021).

Beilinson et al. (2017) pointed out that the incision of this valley occurred before the Last Interglacial. The fluvial-alluvial nature of the late Cenozoic deposits of this area was temporarily interrupted by the marine transgression of MIS 5e and ended with the establishment of well-developed lacunar systems.

The CPFS is located in the middle basin of Quequén Salado River (Fig. 1; 38°49′30"S, 60°32′06"W). The first references and descriptions of Pleistocene levels in this area correspond to the studies carried out by Frenguelli (1928). Subsequently, Pardiñas et al. (1996) referred to the existence of deposits corresponding to the Late Pleistocene along the course of this river, which, according to their sedimentary characteristics and the fossil mammal fauna present, in most cases were correlated with the Luján Formation and its different members (Fidalgo et al., 1973; Bargo et al., 1986). The sedimentary sequence presents profiles with heights that vary between 15 and 20 m. The base of the sequence is covered by the current river level. It begins with Unit A, a dark brown to reddish brown clayey siltstone, finely laminated in the basal sector and solid from the middle sector towards the roof, with abundant oxide patinas of iron. Units B and C are dark green, laminated, sandy silts. Unit D is composed of consolidated clayey silts, light green to greenish gray; these deposits were related with an estuarine environment and tentatively assigned to the MIS 5 transgression (Pardiñas et al., 1996; Beilinson et al., 2017). Overlying this level, and arranged on an erosional contact are dark green to olive green fine sandy silts that correspond to Unit E. Unit F is composed of light green silty clay, with abundant calcium carbonate concretions. Unit G is composed of sandy to clayey silts of yellowish-green color. At Puente Oriente (15 km upstream from this site), Bonadonna et al. (1995) reports three standard ^{14}C dating obtained from *Litoridina parchappei* (13.5–11.4 cal ka BP, 20.8–16.8 cal ka BP; 20.9–17.1 cal ka BP, see Table A.1) that may correspond to this level. Finally, Unit H consists of dark gray silty sands.

Unit D includes a very rich vertebrate assemblage, represented by marine (*Pogonias cromis*, *Micropogonias furnieri*) and continental fishes (*Rhamdia sapo*, *Corydoras paleatus*, and *Synbranchus marmoratus*), reptiles (*Hydromedusa tectifera*), and birds (*Anas* sp., *Ciconia maguari*) and several taxa of continental micro- and macromammals (Pardiñas et al., 1996; Bogan et al., 2012; Beilinson et al., 2017). Likewise, Unit G (Alberdi et al., 1989) also includes several macro- and micromammal taxa.

2. Material and methods

2.1. Sampling

The three sites (Salto de Piedra, Campo Spósito, and Cascada del Paleolama) were sampled for ESR, U-series, OSL, and ^{14}C AMS dating.

At Salto de Piedra, two fossil samples, a tooth (labeled FCS-P 130/lab ID: #545) and a humerus (labeled FCS-P 91.1/lab ID: #544), attributed to *Hippidion principale,* were collected from Unit 1, facies 2 (U1-F2) for U-series dating. The absence of sediment associated with #545 precluded the use of the ESR method. An OSL sediment sample (L0/ARG06, Table A.1.) was collected from Unit 2, facies 4 (U2-F4) by hammering an opaque PVC tube into the section, to avoid exposure to sunlight. Further, a rib fragment of *Equus neogeus* (labeled FCSP-280/lab ID: 5095.1.1) was collected from Unit 4, facies 8 (U4-F8) for ^{14}C AMS dating, together with shells of *Chillina parchappi* from the top of the Unit 5 Facies 11 (U5-F11). Two samples of freshwater mollusks (Lab IDs: 5096.1.1 and 5094.1.1) of *Biomphalaria peregrina* from Unit 5 were also dated for ^{14}C AMS.

The Campo Spósito site was sampled from the excavation area located outside the town of San Pedro. A tooth of *Toxodon platensis* (labeled GCF-8/Lab ID: ARG1805) and associated sediment was collected from the "El Tala" unit for U-series and ESR dating purpose.

Sediment from the site Cascada del Paleolama was also sampled for OSL by hammering an opaque PVC tube (Lab ID: L0/ARG02) into the upper portion of the Unit E (*sensu* Pardiñas et al., 1996). A tooth fragment from a *Macraeuchenia patachonica* skull (labeled FCS-PV.305/Lab ID: ARG1803) recovered from "Planchón verde", the top Unit D (*sensu* Pardiñas et al., 1996) was collected for ESR dating, together with the surrounding sediment for dose rate evaluation. Finally, a tooth of *Lama guanicoe* (labeled MH-QEQ-24-01/Lab ID: ARG1804) recovered from

Unit F (*sensu* Pardiñas et al., 1996), was also used for ESR dating. For this sample, the associated sediment needed for dose rate evaluations had been collected from the site several years ago and had remained in storage (labeled and wrapped in foil) at the Museo Municipal de Ciencias Naturales "Vicente Di Martino" (Monte Hermoso, Argentina).

2.2. ^{14}C AMS dating

The new ^{14}C dates were obtained at the AMS technique at the National Center of Accelerators of the University of Sevilla (CSIC, Spain). Prior to AMS measurement, samples must be treated in the lab to isolate the intrinsic carbon content of the sample and transform it to graphite. Shells follow an acid leaching with HCl 0.5M at room temperature to remove the external surface, which may be contaminated. In the case of bone samples, the hydroxyapatite fraction must be removed to recover the proteinaceous material, commonly referred to as "collagen", which is considered to be the most reliable fraction for dating. At CNA we use a modified Longin method (Longin, 1971) followed by ultrafiltration (Higham et al., 2006). The bone is gently crashed and demineralized using HCl 1M. The sample is then neutralized with Milli-QTM water and NaOH 0.2M is added to eliminate further contaminants (Arslanov and Svezhentsev, 1993). A final acid step with HCl 0.1M, adjusting pH to 2–2.5 is used to hydrolyze collagen at 58 °C overnight. Samples are filtered using previously cleaned EzeeTM filters, and solid residues are discarded. Hydrolyzed collagen is then transferred from the EzeeTM filter to a previously cleaned Amicon Ultra-4 Ultracel 30 ultrafilter, which can retain molecules over 30 kDa. The retained collagen is freeze-dried overnight. Collagen yield and elemental composition of the collagen are checked to ensure the quality of the material (van Klinken, 1999).

The last procedure before AMS measurement is to transform the sample into graphite. At CNA 1 mg of carbon is used for the AMS measurement. Thus, the amount of clean and dry samples used in the graphitization process depends on the concentration of carbon in the material. In the case of calcium carbonate samples, about 15 mg are used, and for collagen, 5 mg. Clean and dry samples are graphitized in an AGE system (Wacker et al., 2010a), which transforms CO_2 obtained from the sample to graphite, which is then pressed into a holder and is ready for AMS measurement. Production of CO_2 depends on the kind of sample. For carbonate samples, a Carbonate Handling System, CHS (Wacker et al., 2013), is used. Carbonate is dissolved using H3PO4 85%, and gas is transferred to the corresponding reactor in AGE. The rest of the organic materials are combusted in an Elemental Analyzer, EA (Elemental Various Micro Cube). When CO_2 is released from EA it is transported to the reactor in AGE, where CO_2 is reduced to graphite, ready to be pressed and used for AMS measurement.

Samples are AMS measured in a Micadas system (Wacker et al., 2010b). A beam of negative ions is produced from the sample, and ^{14}C is selected from the rest of the components of the beam using kinematic filters. To achieve the necessary sensitivity, molecules are broken in the stripper tube at the high voltage terminal. All three isotopes of carbon are measured (^{12}C, ^{13}C, and ^{14}C) in unknown samples, together with standards and background samples, thus obtaining the necessary $^{14}C/^{12}C$ and $^{13}C/^{12}C$ ratios to calculate both $\delta^{13}C$ and radiocarbon age (Age BP). All the data from the measurement are analyzed by BATS software (Wacker et al., 2010c). Radiocarbon ages were calibrated using OxCal V4.4.4 with the corresponding SHCal20 calibration curve (Hogg et al., 2020).

2.3. U-series dating

Laser ablation (LA) and Solution U-series analyses were carried out using a Nu Plasma HR MC-ICP-MS in the Radiogenic Isotope Facility (RIF) at the School of Earth and Environmental Sciences, the University of Queensland (Australia).

2.3.1. LA U-series dating

The LA analyses were performed on samples #544 and #545 from U1 of Salto de Piedra site following the principles and procedures modified after Grün et al. (2014). The LA transects are composed of several rasters performed across a section of the samples. Rather than applying off-line reduction of raw isotope signal intensity data to calculate the sample's $^{230}Th/^{238}U$ and $^{234}U/^{238}U$ activity ratios (or using third-party software such as Iolite for such purpose), we used the MC-ICP-MS online data acquisition software designed for solution analysis to generate baseline fully corrected isotope ratio data directly from the mass spectrometer. Then the $^{230}Th/^{238}U$ activity ratios of the samples were normalized to a homogeneous rhino tooth standard (used in Grün et al., 2014) by interpolating through the results of the standard that was repeatedly measured at the beginning and then after every five unknown samples. The $^{230}Th/^{238}U$ activity ratio of this standard has been precisely calibrated by isotope dilution. This protocol is somehow analogous to micro-drilling of a series of spots along a tooth cross-section for solution MC-ICP-MS dating reported in previous publications (e.g., Price et al., 2013, 2017). The difference is that the laser-ablation protocol does not involve any chemical separation of U and Th.

2.3.2. Solution U-series analyses of dental tissues

Solution U-series analyses were performed on the powdered enamel and dentine tissues resulting from the ESR dating sample preparation procedure (section 2.4), following chemical treatment procedures and MC-ICP-MS analytical protocols described elsewhere (e.g., Clark et al., 2014; Zhao et al., 2009). Powdered sub-samples weighing ~10 mg were spiked with a mixed ^{229}Th-^{233}U tracer and then wholly dissolved in concentrated HNO_3. After digestion, each sample was treated with H_2O_2 to decompose trace amounts of organic matter and to facilitate complete sample-tracer homogenization. U and Th were separated using conventional anion-exchange column chemistry using Bio-Rad AG 1-X8 resin. After stripping off the matrix from the column using double-distilled 7N HNO_3 as eluent, 3 ml of a 2% HNO_3 solution mixed with a trace amount of HF was used to elute both U and Th into a 3.5-ml pre-cleaned test tube. After column chemistry, the U-Th mixed solution was injected into the MC-ICP-MS through a DSN-100 desolvation nebulizer system with an uptake rate of around 0.07 ml per minute. U-Th isotopic ratio measurement was performed on the MC-ICP-MS using a detector configuration to allow simultaneous measurements of both U and Th. Closed-system U-series ages were calculated using the Isoplot/Ex 3.75 Program (Ludwig, 2003).

2.4. Combined U-series/ESR dating

Three fossil teeth (ARG1803 and ARG1804 from Cascada de Paleolama, and ARG1805 from Campo Spósito) were prepared following a standard procedure in ESR dating: the enamel layer was mechanically separated from the other dental tissues and both inner and outer surfaces were removed with a dentist drill to eliminate the volume that received an external alpha dose. The dentine attached to the enamel layer was kept aside for subsequent solution bulk U-series analyses (section 2.3.2). Enamel and dentine were ground and sieved <200 μm. Two samples A and B were prepared from tooth ARG1805.

Dose evaluation utilized the multiple aliquot additive dose (MAAD) method. The enamel powder of each sample was split into eleven aliquots and gamma-irradiated at CENIEH (Spain) with a Gammacell 1000 Cs-137 gamma source (dose rate = 6.27 ± 0.14 Gy/min) to the following doses: 49.9, 99.9, 149.7, 249.6, 349.3, 499.0, 698.7, 898.4, 1497.2 and 2489.0 Gy. One aliquot was kept untouched (=natural aliquot). Room temperature ESR measurements were carried out at CENIEH with an

EMXmicro 6/1 Bruker ESR spectrometer coupled to a standard rectangular ER 4102ST cavity. The following procedure was used to minimize the analytical uncertainties: (i) all aliquots of a given sample were carefully weighted into their corresponding tubes, and a variation of <1 mg was tolerated between aliquots; (ii) ESR measurements were performed using a Teflon sample tube holder inserted from the bottom of the cavity to ensure that the vertical position of the tubes remains exactly the same for all aliquots. The following acquisition parameters were used: 1–12 scans, 1 mW microwave power, 1024 points resolution, 15 mT sweep width, 100 kHz modulation frequency, 0.1 mT modulation amplitude, 20 ms conversion time, and 5 ms time constant. All aliquots were successively measured within a short time interval (<1 h). This procedure was repeated two times over successive days without removing the enamel from the ESR tubes between measurements to evaluate measurement and equivalent dose (D_E) precisions.

The ESR intensities were extracted from the T1-B2 peak-to-peak amplitudes of the ESR signal (Grün, 2000a), and then normalized to the corresponding number of scans and aliquot mass. D_E values were obtained by fitting a single saturating exponential (SSE) through the mean ESR intensities derived from the repeated measurements. Fitting was performed with Microcal OriginPro 9.1 software, which is based on a Levenberg-Marquardt algorithm by chi-square minimization. Data were weighted by the inverse of the squared ESR intensity ($1/I^2$) and the inverse of the squared experimental errors ($1/s^2$). The final 1-σ D_E error used for age calculation includes both the fitting and gamma source dose rate errors.

The environmental dose rate from the sediment (gamma and beta dose rate components) was evaluated from the laboratory analysis of sediment samples collected either in situ or from the fossil specimen itself. No in situ gamma dosimetry was performed. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analyses were performed on all sediment samples by Genalysis Laboratory Services, following a four-acid digest preparation procedure. The following parameters were used for the dose rate calculations: an alpha efficiency of 0.13 ± 0.02 (Grün and Katzenberger-Apel, 1994), Monte-Carlo beta attenuation factors from Marsh (1999), dose-rate conversion factors from Guérin et al. (2011), an estimated water content of 0 and 5 ± 3 wt% in enamel and dentine, respectively. Current water content was measured by drying the sediment in an oven at 40 °C for three weeks. A sample geometry sediment/enamel/dentine was considered for the beta dose rate attenuations. Age calculations were performed with two programs: (i) DATA (Grün, 2009), a DOS-based program using the US (Grün et al., 1988) and CSUS (Grün, 2000b) models, and (ii) USESR, a Matlab-based program (Shao et al., 2014) using the US and AU (Shao et al., 2012) models.

2.5. OSL measurements

Measurements were performed using TL-DA-15 readers fitted with an EM19235QA photomultiplier, 6 mm thick U-340 filters, and a 90Sr/90Y beta source. Equivalent dose (D_e) estimates were determined using the single aliquot regenerative dose (SAR) protocol (Wintle and Murray, 2006). A regenerative and test dose preheat combination of 240 °C for 10 s and 200 °C for 10 s, respectively, was used for sample L021/ARG02 (Cascada de Paleolama). The corresponding preheat combination used for sample L021/ARG06 (Salto de Piedra) was 260 °C for 10 s and 200 °C for 10 s. OSL measurements were made at 130 °C for 50 s. Net OSL values were calculated from the first 0.25 s of stimulation and a background subtraction of the last 12.5 s. Aliquots were rejected if recycling and IR-depletion (Duller, 2003) ratios lay outside 1.0 ± 0.1 and if the percentage of recuperation exceeded 5% (Murray and Wintle, 2000). Dose-response curves were fitted using an exponential plus linear function and D_e values were calculated after interpolating the sensitivity-corrected natural signal onto the dose-response curve in Analyst (Duller, 2015). Mean D_e values have been calculated using the central age model (CAM) of Galbraith et al. (1999). Errors in final ages are presented at 1σ.

Environmental dose rates have been calculated from U, Th and K concentrations determined from sediment sub-samples using ICP-MS (Inductively Coupled Plasma Mass Spectrometry). The conversion factors of Guérin et al. (2011) have been used to calculate external beta and gamma dose rates. Beta dose rates have been corrected for grainsize and etching attenuation (Brennan, 2003). For both samples, it is assumed that the ^{238}U and ^{232}Th series are in secular radioactive equilibrium. A moisture content of 10 ± 2% (expressed as percentage of wet weight) has been used for both samples.

2.6. Faunal collections

Fossil vertebrates recovered from the three sites studied are hosted in collections of different institutions from Argentina: Salto de Piedra at INCUAPA (Olavarría, Buenos Aires Province), Campo Spósito at Museo Paleontológico de San Pedro "Fray Manuel de Torres" (San Pedro, Buenos Aires Province), and Cascada de Paleolama at Museo de La Plata (La Plata, Buenos Aires Province) and Museo Municipal de Ciencias Naturales "Vicente Di Martino" (Monte Hermoso, Buenos Aires Province).

We refined and updated the faunistic lists for each fossil site based on bibliographic review, diagnostic anatomical features, measurements, and the analysis of unpublished collections. Our revisions are also based on unpublished materials recovered after the year of previous publications (Pardiñas et al., 1996; Alberdi et al., 1989; Prado and Alberdi, 2011; Prado et al., 2012). The collection from Salto de Piedra were obtained from systematic excavations performed in the last three years using a terrestrial grid, recording the coordinates of macrovertebrate fossil remains with a Total Station, and wet sieving the sediments to obtain microvertebrates, malacological and entomological remains. The taxonomic identifications from this fossil site are currently in progress.

The aim of a revision of the fossil collections by the same taxonomic specialists, following the zoological record standards of the different taxonomic groups, intends to obtain a homogeneous and highly rigorous faunal list in the case that any of the species previously identified could yield the minimum ambiguity for the first appearance or index fossils of the biozones.

3. Results

3.1. Radiocarbon dating

Radiocarbon results obtained from Salto de Piedra are shown in Table 2, following the conventions in Millard (2014). All $\delta^{13}C$ values have been calculated using AMS ratios, so they do not correspond to the original sample, but to the graphite produced from the sample. From the bone samples, one of them (SP-5, CNA5097.1.1) did not pass the quality checks since collagen yield was only 0.13%, and the minimum acceptable is 0.5%. The other bone sample, SP-3 (CNA5095.1.1), had a collagen yield of 3.54%, and the weight concentration of carbon and the carbon and nitrogen weight concentration in the collagen was 39.6% and 14.3% respectively, leading to an atomic C:N ratio of 3.2. All these quality parameters are representative of very good-quality collagen (Van Klinken,1999).

For the following discussion of the results, all the possible calibrated age ranges in the radiocarbon samples with more than one will be merged into only one range from the youngest to the oldest possible age