

Multidisciplinary insights into the Late Pleistocene-Holocene palaeoenvironment at the archaeo-palaeontological Loltún Cave site in Yucatán, Mexico

Supplementary Information

1. Background

Research at the Loltún cave has confirmed its exceptional significance for both Neotropical palaeoecology and Maya archaeology. It stands as one of the few sites in the region, and the first on the Yucatán Peninsula, to preserve a long-term sedimentary sequence. This is remarkable given that the regional geomorphology usually hinders the accumulation and preservation of sediments and organic matter. This unique geological archive has yielded a rich Pleistocene fauna assemblage in stratigraphic association with stone tools, providing critical data for palaeoecological reconstruction and the study of early human subsistence strategies. Crucially, the cave's stratified deposits also provide a continuous record of human occupation, documenting pre-ceramic materials and a complete cultural sequence from the Preclassic throughout the Postclassic periods. By integrating this archaeological evidence with palaeobiological data provides unparalleled insight into the interplay between environmental conditions and social evolution, illuminating the foundational landscape and cultural dynamics that underpinned the development of the Maya civilisation over millennia (Thompson, 1897; Mercer, 1896; Álvarez & Polaco, 1972; Konieczna, 1981; González-Lincon, 1986; Xelhuantzi & López, 1986; Velázquez & Valadez, 1978, 1980; Arroyo-Cabrales & Álvarez, 2003; Morales-Mejía, 2009; Gutiérrez-García et al., 2015, 2021; Cruz-Silva et al., 2016). Despite their potential, previous excavations conducted at the site in the 20th century did not yield conclusive data to clarify questions about the early human presence in the peninsula. Furthermore, past excavation procedures resulted in vague explanations of the formation processes of this unique deposit, as well as partial and incomplete reconstructions of the region's cultural evolution (Velázquez-Valadez, 1980a).

2. Studied area details

The Puuc region, meaning 'hill' in Mayan, is a distinct geographical and cultural area bounded by the Sierrita de Ticul escarpment, extending from Maxcanú to Tzucacab and Dzilbalché

(Barrera Rubio, 1995). This region contains the Yucatán Peninsula's highest elevations. Its karstic landscape features numerous caves, with some containing permanent water sources and others, like Loltún, remaining dry or experiencing only seasonal flooding.

Loltún Cave (20°15.28' N, 89°27.4' W), at 90 masl, is an 850 x 500-meter east-west oriented system with chambers connected by passages. Roof collapses created large openings, including the Loltún and Nahkab entrances (Fig. S1).

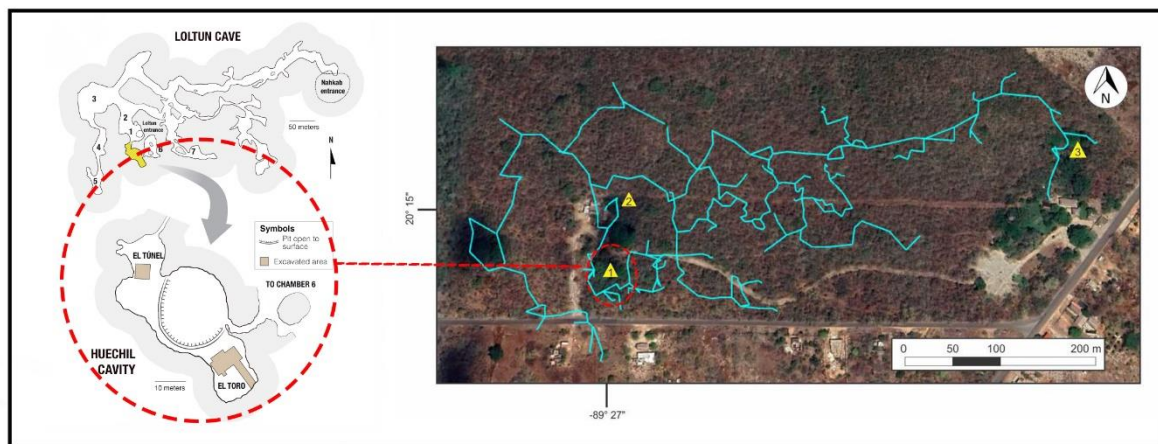


Figure S1. Polygonal survey of Loltún Cave 2024. An aerial image (right) provides regional context for the cave system. Triangular markers indicate the entrances to the cave system: 1) Huechil chamber, 2) Loltún, and 3) Nahkab.

Late 19th Century: The first scientific excavations were conducted in 1889 by Edward H. Thompson (Peabody Museum, Harvard), who recovered ceramics, lithics, faunal remains, and documented the 'Warrior of Loltún' bas-relief (Thompson, 1897). Henry C. Mercer (University of Pennsylvania) later systematically examined 29 caves nearby (Mercer, 1896).

Late 20th Century: The 1977-1980 Loltún Project by INAH, directed by Norberto González Crespo with fieldwork by Ricardo Velázquez Valadez, conducted the first systematic excavations. Work in the Nahkab chamber yielded Maya ceramics, while excavations at the Huechil chamber's rock shelters (El Túnel and El Toro) uncovered significant Preceramic cultural deposits, providing groundbreaking evidence for early human occupation (Velázquez Valadez, 1978, 1980a, b, c; González-Licón, 1986).

The Huechil Chamber, accessible via a narrow passage beneath Loltún Cave's entrance (named from the Mayan term “huech” denoting armadillo), constitutes a significant karstic

feature measuring approximately 30 meters in diameter with a prominent ceiling opening and 10-meter vertical drop forming a debris cone (Velázquez Valadez, 1980a). Systematic excavations of the adjacent El Túnel and El Toro rock shelters (1977-1980) revealed the site's integrity, culminating in the 1978 discovery of a gomphothere skeleton in El Toro's deeper layers (reaching 10 m depth) under the supervision of INAH researchers (Velázquez-Valadez, 1980b, 1980c) (Fig. S2). The site's exceptional preservation has yielded 68 mammalian taxa, including extinct Pleistocene megafauna (*Equus* sp. and *Cuvieronius* sp.) and extant species. Ancient DNA analysis of *Ototylomys phyllotis* rodents has demonstrated 500,000 years of genetic continuity (Gutiérrez-García et al., 2015)

Archaeological evidence at the time suggested that human occupation of the area spanned at least 9,000 yr. BP, beginning with Late Pleistocene hunter-gatherer groups, as evidenced by lithic assemblages in the lower units at El Toro rock shelter (Velázquez-Valadez, 1980 a,b,c). This occupational sequence progresses through the emergence of sedentary Maya communities during the Early Preclassic period (radiocarbon age of 1840 ± 105 yrs. BP) (Velázquez-Valadez, 1978), dating the introduction of ceramics to the northern part of the peninsula to around 1500–1600 BP (Velázquez-Valadez, 1978), culminating in the radical transformation of indigenous socioecological systems during the Colonial period. However, stratigraphic complexities have been unresolved, particularly the apparent chronological overlap between ceramic-phase deposits and terminal Pleistocene megafauna remains (Arroyo-Cabrales and Álvarez, 2003). Additionally, palaeoenvironmental reconstructions have been derived from analyses of fossil amphibians and reptiles collected during excavations conducted between 1970 and 1980 excavations, providing data for understanding the Late Pleistocene climatic conditions and ecological dynamics in the region (Arroyo-Cabrales and Álvarez, 2003; Cruz-Silva et al., 2016).

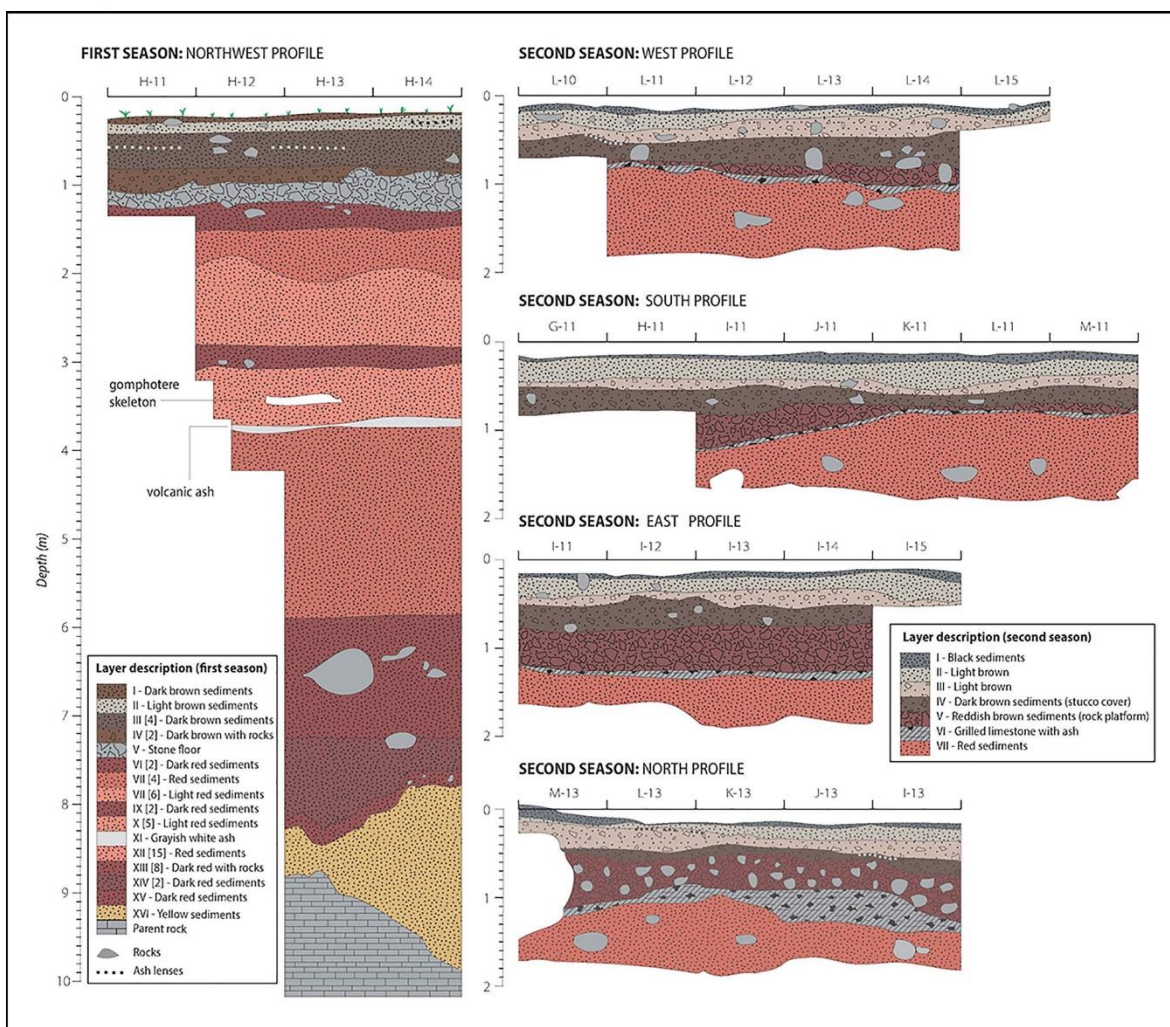


Figure S2. Stratigraphic profiles from excavations conducted during the first (1970s) and second (1980s) field seasons. (Adapted from Velázquez-Valadez, 1978, 1980b.)

3. Project execution specifications

3.1 Fieldwork specifications for project execution

The Loltún Archaeological Project is a multidisciplinary initiative investigating long-term human-environment dynamics on the Yucatán Peninsula. As part of this program, a 2024 field season involved a systematic excavation at the Huechil Chamber within the "El Toro" rock shelter (see Fig. S3). This location was previously excavated in 1978 to a depth of nearly ten metres, reaching bedrock. Fieldwork carried out during these seasons revealed that the site boasts exceptional stratigraphic integrity, preserving a cultural and palaeobiological sequence that spans from the late Pleistocene to the recent Holocene. A key finding was the presence of stone tools and remains of extinct megafauna in the same stratigraphic levels. The sequence was further documented by a distinctive ash layer that stratigraphically separates two primary faunal accumulations from the Late Pleistocene. Despite the valuable contributions of this earlier work, key questions remain unresolved. These include the origin and chronology of the volcanic ash layer, as well as the nature of the association between Pleistocene faunal remains and ceramics from the Maya occupation period. The excavation reported here aimed to re-evaluate the stratigraphic integrity of the site and enable a comprehensive analysis of palaeobiological data across the different phases recorded in this section of the cave.

Our research priorities focused on evaluating the vertical distribution of materials and reconstructing the long-term palaeoenvironment through a combination of field observations and novel analytical techniques. This included the use of plant wax biomarkers, stable isotope analysis ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$ and $\delta^{15}\text{N}$), zooarchaeology by mass spectrometry (ZooMS), geochemical (XRF) and magnetic susceptibility analysis, as well as radiocarbon dating. All 71 analysed samples by technique are shown in the comprehensive Table S1, recovered materials were transported to the Subdirección de Arqueología Subacuática (INAH-SAS) of the Instituto Nacional de Antropología e Historia (INAH) in Campeche, Mexico, for analysis.

A systematic 1×1 m square grid was established, attempting to maintain a pre-existing alphanumeric coordinate system. Squares were numbered sequentially from 15 (northern extent) to 10 (southern extent), and lettered from E (west) to K (east). Each square was assigned a unique identifier (e.g., H12). This standardised referencing system ensured consistency with prior protocols and facilitated spatial analysis. The primary datum, initially

set 10 cm above the modern surface, was subsequently lowered and fixed to a grid established at a depth of 1.5 m. This adjustment facilitated more accurate measurement of deeper contexts and aligned the recording system with the top of the paleontological deposit (Layer VI). Stratigraphic units were characterized in the field based on sediment texture and Munsell colour. The excavation was more detailed in squares that had not been previously excavated, from E15 to G15 and from E15 to E12, corresponding to the northeast perimeter. This involved excavating in arbitrary 10 cm spits, with plan maps and detailed context sheets documented for each level. Conversely, in squares G13 and G14—an area previously excavated in the 1970s to depths of nearly 10 m.—the methodology focused on rapid cleaning and profiling of the existing excavation walls to expose and record intact stratigraphic sequences for correlation with the new sectors.

Work commenced at the edge of an existing southeast-northwest oriented trench. An 8 m² area surrounding this location was cleaned, and the southern profile of the earlier excavation was exposed in squares 10I–10J (see Fig. S4). Intervention was deliberately limited to a maximum depth of 1.8 metres from the primary datum (see Fig. S5). After profiling, excavation proceeded in the area comprising squares E15 to E12 and H15 to H12. A deep test pit was excavated between squares F13–F14 and G13–G14 to sample deeper stratigraphy (see Fig S6).

Samples were collected in the field except for the sequential stable carbon and oxygen isotope analysis, for which subsamples were taken at the MPI-GEA (LTN-5-1 to LTN-5-12). Almost complete molars were submitted for radiocarbon dating. Of these, two specimens — LTN-8 (UGAMS#73926), corresponding to a camelid (*Hemauchenia macrocephala*), and LTN-9 (UGAMS#73927), corresponding to a horse (*Equus* sp.) — were subsampled for stable carbon and oxygen isotope analysis, yielding samples LTN-2 and LTN-3. Thirty-six sediment samples for biomarker analysis (LTN-22 to LTN-57) were collected contiguously at 10 cm intervals. Additionally, eleven sediment samples (LTN-11 to LTN-21) corresponding to representative changes in the colour of the stratigraphic sequence were collected for analysis of major and trace elements, as well as magnetic susceptibility.

Sampling for biomarker, geochemical and magnetic susceptibility analyses began at the deepest point of the eastern profile of the unexcavated square, F13. After cleaning and levelling the surface of the southern profile of square I10, sampling continued upwards until

reaching the modern ground surface (see Fig. S5 and S8). Thirteen bone fragments for ZooMS and stable nitrogen isotope analysis were randomly selected from Pleistocene strata. Most were non-diagnostic; exceptions include a probable feline metacarpal (LTN-68), a sacrum from a medium-sized animal (LTN-69) and a proximal metacarpal epiphysis from a horse (LTN-58).

Table S1. Analysed samples by technique and procedence								
Provenience	Sample number	Laboratory code	Technique	Material type	Taxa	Layer	Depth below datum (m)	Archaeological context according to the layer classification of Velázquez-Valadez (1980).
El Toro	LTN-1	LTN-002	Stable Isotope Analysis ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$, $\delta^{15}\text{N}$) and ZooMS	Tooth Enamel	<i>Equus</i> sp.	VI - VII	1.6 - 2	Lower Preclassic/Preceramic
El Toro	LTN-2	LTN-003	Stable Isotope Analysis ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$, $\delta^{15}\text{N}$) and ZooMS	Tooth Enamel	<i>Hemauchenia macrocephala</i>	IX - XIII	2.6 - 3.3	Preceramic
El Toro	LTN-3	LTN-004	Stable Isotope Analysis ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$, $\delta^{15}\text{N}$) and ZooMS	Tooth Enamel	<i>Equus</i> sp.	VI	1.7	Mid Preclassic / Lower Preclassic
El Toro	LTN-4	LTN-005	Stable Isotope Analysis ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$, $\delta^{15}\text{N}$) and ZooMS	Tooth Enamel	<i>Equus</i> sp.	XII	3.1	Preceramic
El Toro	LTN-5	LTN-005 (seq.)	Stable Isotope Analysis ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$, $\delta^{15}\text{N}$) and ZooMS	Tooth Enamel	<i>Equus</i> sp.	XII	3.1	Preceramic
El Toro	LTN-6	UGAMS#73924	14C radiocarbon and $\delta^{13}\text{C}$ values	Enamel bioapatite	<i>Equus</i> sp.	VI - VII	1.6 - 2	Mixed
El Toro	LTN-7	UGAMS#73925	14C radiocarbon and $\delta^{13}\text{C}$ values	Enamel bioapatite	<i>Equus</i> sp.	XII	3.1	Preceramic
El Toro	LTN-8	UGAMS#73926	14C radiocarbon and $\delta^{13}\text{C}$ values	Enamel bioapatite	<i>Hemauchenia macrocephala</i>	XIII	2.6 - 3.3	Preceramic
El Toro	LTN-9	UGAMS#73927	14C radiocarbon and $\delta^{13}\text{C}$ values	Enamel bioapatite	<i>Equus</i> sp.	VI	1.7	Mid Preclassic / Lower Preclassic
El Toro	LTN-10	UGAMS#73928	14C radiocarbon and $\delta^{13}\text{C}$ values	Enamel bioapatite	<i>Equus</i> sp.	VII	1.9	Lower Preclassic
El Toro	LTN-11	L0L-H1	XRF and MS	Sediment		XV	3.7	Preceramic
El Toro	LTN-12	L0L-H2	XRF and MS	Sediment		XIII	3.3	Preceramic
El Toro	LTN-13	L0L-H3	XRF and MS	Sediment		XII	3	Preceramic
El Toro	LTN-14	L0L-H4	XRF and MS	Sediment		X	2.8	Preceramic
El Toro	LTN-15	L0L-H5	XRF and MS	Sediment		X	2.6	Preceramic
El Toro	LTN-16	L0L-H6	XRF and MS	Sediment		VIII	2.2	Preceramic
El Toro	LTN-17	L0L-H7	XRF and MS	Sediment		VII	1.9	Lower Preclassic
El Toro	LTN-18	L0L-H8	XRF and MS	Sediment		VI	1.7	Mid Preclassic / Lower Preclassic
El Toro	LTN-19	L0L-H9	XRF and MS	Sediment		V	1.3	Mit Preclassic
El Toro	LTN-20	L0L-H10	XRF and MS	Sediment		IV	0.6	Early classic / Upper Preclassic
El Toro	LTN-21	L0L-H11	XRF and MS	Sediment		II	0.3	Late classic / Early classic
El Toro	LTN-22	LTN-0018	Plant Wax Biomarker Proxies	Sediment		XV	3.6	No material culture
El Toro	LTN-23	LTN-0019	Plant Wax Biomarker Proxies	Sediment		XIII	3.5	No material culture
El Toro	LTN-24	LTN-0020	Plant Wax Biomarker Proxies	Sediment		XIII	3.4	No material culture
El Toro	LTN-25	LTN-0021	Plant Wax Biomarker Proxies	Sediment		XIII	3.3	No material culture
El Toro	LTN-26	LTN-0022	Plant Wax Biomarker Proxies	Sediment		XII	3.2	Preceramic
El Toro	LTN-27	LTN-0023	Plant Wax Biomarker Proxies	Sediment		XII	3.1	Preceramic
El Toro	LTN-28	LTN-0024	Plant Wax Biomarker Proxies	Sediment		XI	3	No material culture
El Toro	LTN-29	LTN-0025	Plant Wax Biomarker Proxies	Sediment		X	2.9	No material culture
El Toro	LTN-30	LTN-0026	Plant Wax Biomarker Proxies	Sediment		IX	2.8	No material culture
El Toro	LTN-31	LTN-0027	Plant Wax Biomarker Proxies	Sediment		IX	2.7	Preceramic
El Toro	LTN-32	LTN-0028	Plant Wax Biomarker Proxies	Sediment		IX	2.6	Preceramic
El Toro	LTN-33	LTN-0029	Plant Wax Biomarker Proxies	Sediment		IX	2.5	Preceramic
El Toro	LTN-34	LTN-0030	Plant Wax Biomarker Proxies	Sediment		VIII	2.4	Preceramic
El Toro	LTN-35	LTN-0031	Plant Wax Biomarker Proxies	Sediment		VIII	2.3	Preceramic
El Toro	LTN-36	LTN-0032	Plant Wax Biomarker Proxies	Sediment		VII	2.2	Preceramic
El Toro	LTN-37	LTN-0033	Plant Wax Biomarker Proxies	Sediment		VII	2.1	Preceramic
El Toro	LTN-38	LTN-0034	Plant Wax Biomarker Proxies	Sediment		VII	2	Preceramic
El Toro	LTN-39	LTN-0035	Plant Wax Biomarker Proxies	Sediment		VII	1.9	Lower Preclassic
El Toro	LTN-40	LTN-0036	Plant Wax Biomarker Proxies	Sediment		VI	1.8	Lower Preclassic
El Toro	LTN-41	LTN-0037	Plant Wax Biomarker Proxies	Sediment		VI	1.7	Mid Preclassic / Lower Preclassic
El Toro	LTN-42	LTN-0038	Plant Wax Biomarker Proxies	Sediment		VI	1.6	Mid Preclassic / Lower Preclassic
El Toro	LTN-43	LTN-0039	Plant Wax Biomarker Proxies	Sediment		VI	1.5	Mid Preclassic / Lower Preclassic
El Toro	LTN-44	LTN-0040	Plant Wax Biomarker Proxies	Sediment		VI	1.4	Mit Preclassic
El Toro	LTN-45	LTN-0041	Plant Wax Biomarker Proxies	Sediment		VI	1.3	Mit Preclassic
El Toro	LTN-46	LTN-0042	Plant Wax Biomarker Proxies	Sediment		VI	1.2	Mit Preclassic
El Toro	LTN-47	LTN-0043	Plant Wax Biomarker Proxies	Sediment		VI	1.1	Late Preclassic / Mit Preclassic
El Toro	LTN-48	LTN-0044	Plant Wax Biomarker Proxies	Sediment		VI	1	Upper Preclassic / Mit Preclassic
El Toro	LTN-49	LTN-0045	Plant Wax Biomarker Proxies	Sediment		IV	0.9	Upper Preclassic / Mit Preclassic
El Toro	LTN-50	LTN-0046	Plant Wax Biomarker Proxies	Sediment		IV	0.8	Upper Preclassic / Mit Preclassic
El Toro	LTN-51	LTN-0047	Plant Wax Biomarker Proxies	Sediment		IV	0.7	Early classic / Upper Preclassic
El Toro	LTN-52	LTN-0048	Plant Wax Biomarker Proxies	Sediment		IV	0.6	Early classic / Upper Preclassic
El Toro	LTN-53	LTN-0049	Plant Wax Biomarker Proxies	Sediment		III	0.5	Late classic/ Early classic / Preclassic
El Toro	LTN-54	LTN-0050	Plant Wax Biomarker Proxies	Sediment		III	0.4	Late classic/ Early classic / Preclassic
El Toro	LTN-55	LTN-0051	Plant Wax Biomarker Proxies	Sediment		II	0.3	Late classic / Early classic
El Toro	LTN-56	LTN-0052	Plant Wax Biomarker Proxies	Sediment		I	0.2	Late Classic
El Toro	LTN-57	LTN-0053	Plant Wax Biomarker Proxies	Sediment		I	0.1	Late Classic
El Toro	LTN-58	LTN-0001	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	<i>Equus</i> sp.	VI	1.8	Lower Preclassic
El Toro	LTN-59	LTN-0006	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	IX	2.8	No material culture
El Toro	LTN-60	LTN-0007	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	VII	2.2	Preceramic
El Toro	LTN-61	LTN-0008	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	XII	3.5-3.6	No material culture
El Toro	LTN-62	LTN-0009	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	VII	2-2.2	Preceramic
El Toro	LTN-63	LTN-0010	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	VI/VII	1.7-2	Mixed
El Toro	LTN-64	LTN-0011	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	IX	2.6	Preceramic
El Toro	LTN-65	LTN-0012	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	XIII/XV	3.3-3.6	No material culture
El Toro	LTN-66	LTN-0013	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	VII	1.9-2	Lower ceramic/Preceramic
El Toro	LTN-67	LTN-0014	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	na	na	na
El Toro	LTN-68	LTN-0015	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	Felidae	VI-VII	1.7-2.2	Mixed
El Toro	LTN-69	LTN-0016	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	VI-VII	1.7-2.2	Mixed
El Toro	LTN-70	LTN-0017	Stable Isotope Analysis ($\delta^{15}\text{N}$) and ZooMS	Bone fragment	None	VI-VII	1.70-2.20	Mixed

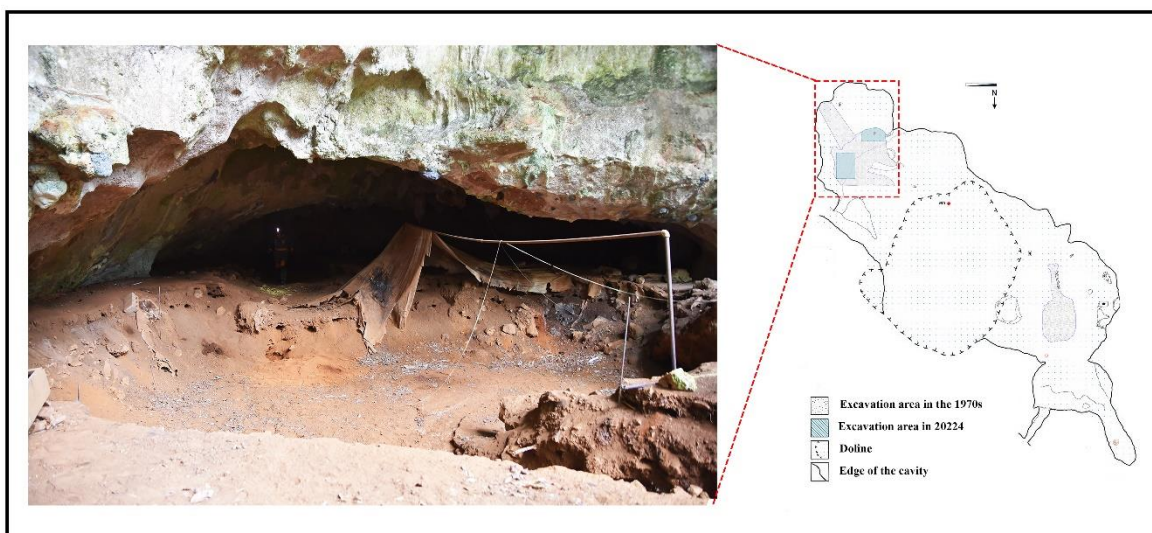


Figure S3. The El Toro rockshelter prior to excavation. The photograph shows the site's condition before the area was prepared for excavation. The updated planimetry of the Huechil chamber is shown on the right.

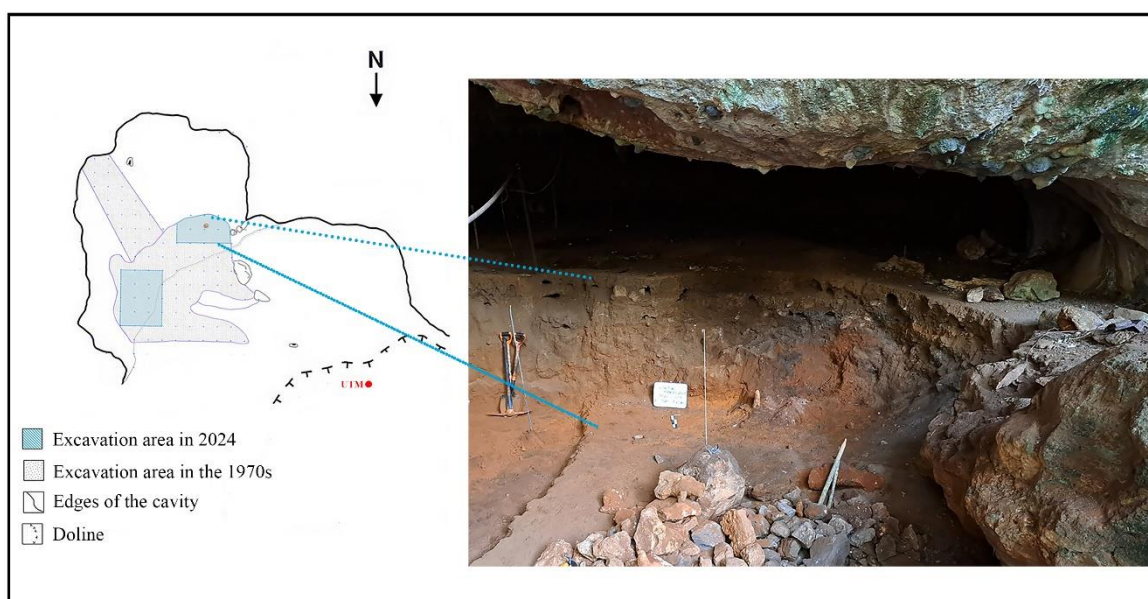


Figure S4: The photograph shows the intervened area to expose the southern profile of the excavation area. This profile is located in squares I10 and J10, next to the trench from the 1970s excavation, which is oriented towards the south-west and inside the rock shelter.

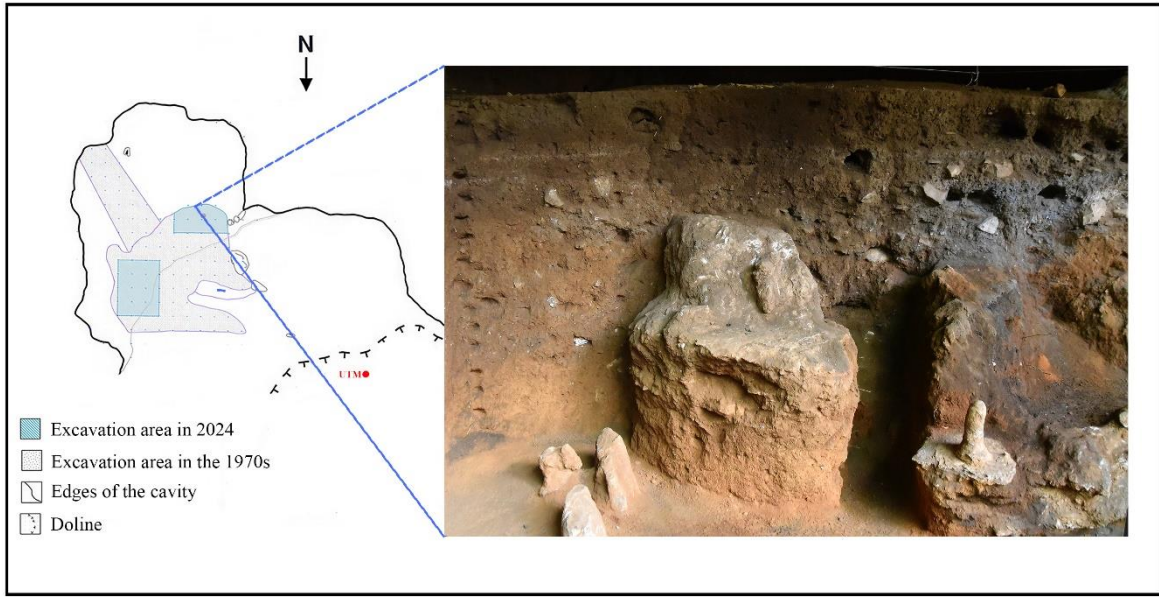


Figure S5: Photograph of the southern profile of excavation square I10, located just west of the 1970s excavation trench. The continuous vertical sequence on the left side indicates the sampling points at 10 cm intervals.

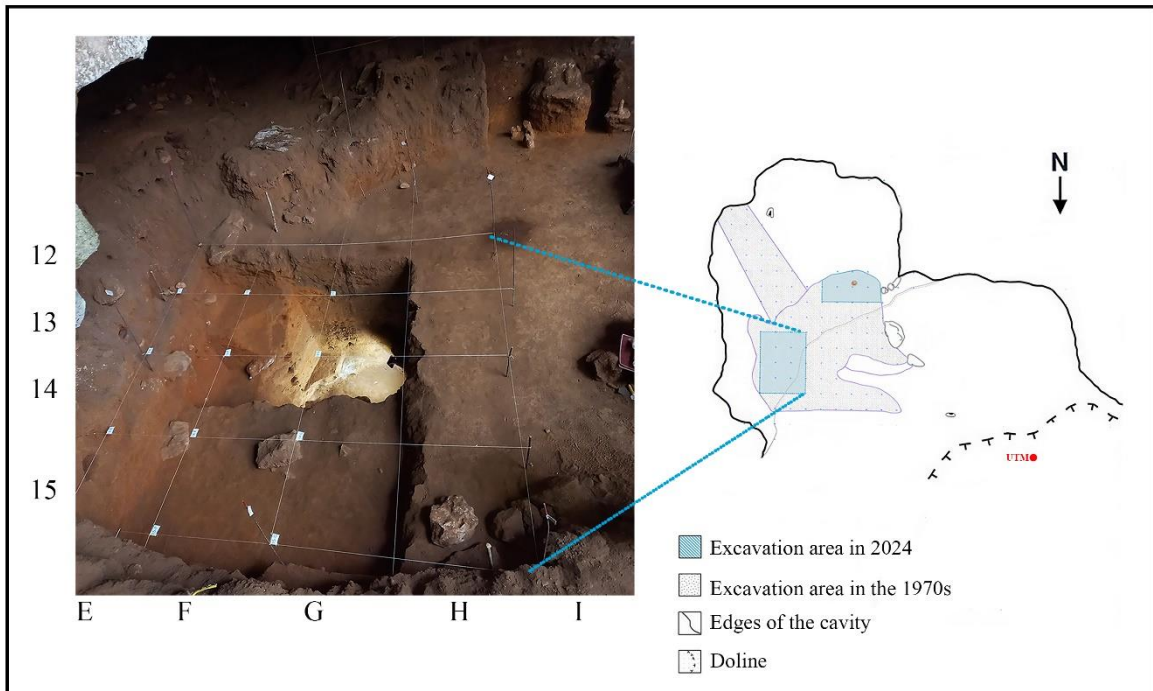


Figure S6: The northeast excavation pit showing the modern grids. On the right is the planimetry of the El Toro rockshelter, overlaying the excavation areas from the 1970s and the 2024 campaign.

3.2 Laboratory specifications for project execution

Table S1 summarizes the main characteristics, provenance, and analytical techniques employed for a total of 71 samples. These include all specimens that successfully yielded results from the various analyses conducted.

¹⁴C AMS dating:

Due to unacceptable collagen preservation in all five teeth, enamel bioapatite was targeted for dating. The process began with a diluted (1N) acetic acid treatment to remove surface-absorbed and secondary carbonates, a step that involved periodic evacuation to ensure evolved CO₂ was removed and that fresh acid permeated the sample's interior micro-surfaces. The chemically cleaned bioapatite was then reacted under vacuum with 100% phosphoric acid to dissolve the mineral matrix and release CO₂. The resulting gas was cryogenically purified and catalytically converted to graphite using the method of Vogel et al. (1984). The ¹⁴C/¹³C ratios of the graphite were measured on the CAIS 0.5 MeV accelerator mass spectrometer, with sample ratios compared to the Oxalic Acid I standard (NBS SRM 4990). Separate $\delta^{13}\text{C}$ values were obtained via stable isotope ratio mass spectrometry with an analytical error of <0.1‰. Conventional radiocarbon ages are reported as uncalibrated years BP (before 1950) using the Libby half-life (5568 years). The $\pm 1\sigma$ error includes both statistical and experimental uncertainties, and a $\delta^{13}\text{C}$ -based fractionation correction was applied. Calibration to calendar years was performed using the IntCal20 calibration curve (Reimer et al., 2020) within the 'clam' package (v.2.4.1) in R, with calibrated age ranges reported as 1σ (68% probability) intervals, rounded to the nearest 10 years.

Stable isotope analysis:

All five specimens (four horse, one llama), curated at the SAS in Campeche, were photographed prior to analysis. The sampling strategy included bulk sampling, where approximately 10 mg of enamel powder was drilled along the full buccal surface of samples LTN-1 to LTN-4 to average isotopic signals over prolonged growth, and sequential sampling, where twelve approximately 10 mg samples (LTN-5-1 to LTN-5-12) were drilled along the crown growth axis from cusp to cervix, avoiding the occlusal surface and enamel-root

junction by about 2 mm, with each sample path measuring approximately 1 mm wide by 3 mm deep. At the MPI-GEA, fragments were cleaned via air-abrasion, and enamel powder was then treated with 1 mL of 0.1 M acetic acid for 10 minutes, followed by three ultra-pure water rinses (Lee-Thorp et al., 2012; Roberts et al., 2017; Sponheimer et al., 2005). For isotopic measurement, 2 mg of pretreated enamel from each sample was reacted with phosphoric acid, and the evolved CO₂ was analysed using a Thermo GasBench II coupled to a Delta V Advantage mass spectrometer. The resulting $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ values were calibrated against international standards (IAEA-603, IAEA-CO-8, USGS44) and an in-house Merck standard. Machine error, determined from Merck replicates, was $\pm 0.1\text{‰}$ for $\delta^{13}\text{C}$ and $\pm 0.2\text{‰}$ for $\delta^{18}\text{O}$, while overall measurement precision, assessed via a bovid enamel standard ($n = 20$), was $\pm 0.2\text{‰}$ for $\delta^{13}\text{C}$ and $\pm 0.3\text{‰}$ for $\delta^{18}\text{O}$.

Plant Wax Biomarker:

A total of 36 sediment samples from a 370 cm profile recovered from Loltún Cave, Mexico, were analysed at 10 cm resolution. Samples were homogenised using an agate mortar and pestle and dried at 40 °C for 12 h. Approximately 5 g of each dried sample were mixed with Ottawa Sand and loaded into Büchi extraction cells. Lipid extraction was performed using a Büchi SpeedExtractor E-916 pressurised solvent extraction system with dichloromethane:methanol (9:1, v/v) at 100 °C and 103 bar (1500 psi) over three 10-min cycles. Total lipid extracts (TLEs) were concentrated to ~1 mL using a Büchi SyncorePlus evaporator and dried under a gentle stream of high-purity nitrogen. The non-polar fraction was isolated using glass column chromatography packed with activated silica gel (100–200 mesh), pre-heated at 150 °C for 24 h, and eluted with HPLC-grade hexane (MERCK). Unsaturated alkenes were removed using AgNO₃-impregnated silica gel chromatography. The purified *n*-alkane fraction was concentrated to 0.5 mL under nitrogen and transferred to 1.5 mL Agilent vials for analysis. All sample preparation and analytical work was conducted at the Max Planck Institute of Geoanthropology, Jena, Germany. All glassware, quartz wool, and silica gel were pre-combusted at 550 °C for at least 5 h prior to use.

n-Alkane analyses were carried out using an Agilent 7890B gas chromatograph equipped with an HP-5MS capillary column (30 m × 0.25 mm × 0.25 µm) coupled to an Agilent 5977A mass selective detector. Samples were injected in splitless mode at 250 °C.

The oven temperature programme started at 60 °C (1 min hold), increased to 180 °C at 6 °C min⁻¹, then to 310 °C at 10 °C min⁻¹, with a final hold of 12 min. Helium was used as the carrier gas at a constant flow rate of 1.3 mL min⁻¹. The MSD operated in electron ionisation mode (70 eV) with a source temperature of 230 °C and full-scan acquisition from m/z 50 to 550. Compound identification was based on retention times relative to a Supelco C8–C40 *n*-alkane standard mixture. Molecular indices were calculated to characterise *n*-alkane distributions, including Carbon Preference Index (CPI; Marzi et al., 1993), Average Chain Length (ACL; Eglinton and Hamilton, 1967), P_{aq}, Odd–Even Predominance (OEP; Hoefs et al., 2002), and P_{wax} (Hoefs et al., 2002), following published formulations: The CPI was calculated following Marzi et al. (1993) to quantify the odd-over-even predominance of long-chain *n*-alkanes:

$$1) \text{ CPI} = \frac{1}{2} \left[\frac{(C_{25}+C_{27}+C_{29}+C_{31}+C_{33})}{(C_{24}+C_{26}+C_{28}+C_{30}+C_{32})} + \frac{(C_{25}+C_{27}+C_{29}+C_{31}+C_{33})}{(C_{26}+C_{28}+C_{30}+C_{32}+C_{34})} \right]$$

The ACL was calculated following Eglinton and Hamilton (1967) as the weighted mean of long-chain *n*-alkanes:

$$2) \text{ ACL} = \frac{(25 \times C_{25}) + (27 \times C_{27}) + (29 \times C_{29}) + (31 \times C_{31}) + (33 \times C_{33})}{C_{25} + C_{27} + C_{29} + C_{31} + C_{33}}$$

The Long-chain Alkane Ratio (LAR) was used to assess the relative contribution of longer-chain *n*-alkanes and was calculated as:

$$3) \text{ LAR} = \frac{(C_{31} + C_{33})}{(C_{27} + C_{29} + C_{31} + C_{33})}$$

where C_n represents the peak area (or concentration) of the *n*-alkane with *n* carbon atoms.

$$4) \text{ P}_{\text{aq}} = (C_{23} + C_{25}) / (C_{23} + C_{25} + C_{31} + C_{33})$$

$$5) \text{ P}_{\text{wax}} = (C_{27} + C_{29} + C_{31}) / (C_{23} + C_{25} + C_{27} + C_{29} + C_{31})$$

The Odd–Even Predominance (OEP) index was calculated following Hoefs et al. (2002) to assess the degree of *n*-alkane degradation:

$$6) \text{ OEP} = \frac{(C_{25}+C_{27}+C_{29}+C_{31}+C_{33})}{(C_{24}+C_{26}+C_{28}+C_{30}+C_{32})}$$

where C_n represents the peak area (or concentration) of the n -alkane with n^{th} carbon atoms. Lower OEP values indicate reduced odd-over-even predominance and increased degradation of n -alkanes. OEP values below 5 indicate enhanced n -alkane degradation.

These molecular proxies are commonly used to characterise changes in organic matter sources and depositional conditions through time. CPI reflects odd-over-even predominance of long-chain n -alkanes and is sensitive to the preservation state of plant-derived organic matter. ACL of n -alkanes provide information on the relative contribution of short- versus long-chain compounds. P_{aq} expresses the relative abundance of mid-chain n -alkanes compared to long-chain homologues, while P_{wax} reflects the proportion of long-chain plant waxes within the total n -alkane pool. Together, these indices summarise variations in n -alkane composition along the sediment profile.

X-ray fluorescence (XRF)

Major and trace elements concentration were measured using a portable Thermo Scientific Niton XL3t 955 ULTRA Analyzer of energy dispersive X-ray fluorescence (XRF) composed of an X-ray tube of Ag anode of 50 kV and 40 mA, and X-ray detector of High-performance semiconductor. The calibration was made from HISS-1 and MESS-3, commonly used in marine sediments. Fe, Ca, Al, Si, Zr, Sr, Mn, Cr, Ti, and K were analyzed for 180 seconds in mining mode, expressed in weight percentage (wt. %), whereas Ba and Rb elements were analyzed for 120 seconds in soil mode, expressed in parts per million (ppm). The raw data (Table S2) were converted to weight percentages, normalized into log, and analyzed using PAST version 4.03 software (Hammer et al., 2001). To establish a relationship among our variables (geochemical and magnetic susceptibility data), a Principal Component Analysis (PCA) was performed to identify a set of variables related to geochemical processes and environmental proxies (Table S3). Additionally, a Spearman correlation test with a significance $p < 0.05$ was carried out, given the non-parametric nature and small size of the dataset.

Table S2. Raw data of chemical elements concentrations and magnetic properties.																	
SAMPLE	Zr	Sr	Fe	Mn	Cr	Ti	Ca	K	Al	Si	Cl	Rb	Ba	MS	MS-HF	MS-LF	MS-FD
	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %	ppm	ppm	SI	SI	SI	%
35	0.025	0.020	5.111	0.081	0.041	0.282	11.35	0.372	1.33	3.42	0.042	0.0051843	0.0186623	617.3	535	604	11.42
65	0.027	0.023	4.864	0.083	0.036	0.276	13.33	0.430	1.31	3.47	0.047	0.005087	0.0220343	632	536.3	613	12.50
135	0.024	0.020	5.173	0.080	0.037	0.303	8.18	0.406	1.30	3.70	0.051	0.0051067	0.0178107	340.6	313.3	359.66	12.88
175	0.034	0.020	5.976	0.133	0.043	0.318	4.89	0.422	1.35	4.013	0.053	0.005498	0.019581	204	188.3	219.33	14.13
195	0.037	0.022	6.614	0.073	0.045	0.349	3.62	0.417	1.39	3.99	0.063	0.0063277	0.0153723	241	235.3	274.33	14.21
225	0.035	0.021	6.194	0.095	0.039	0.343	4.18	0.37	1.32	3.99	0.066	0.0058157	0.0162327	215.6	191.3	221.66	13.68
265	0.036	0.023	5.993	0.027	0.038	0.309	4.97	0.357	1.26	3.76	0.065	0.005936	0.0146733	210.3	285	331.66	14.07
285	0.032	0.022	5.935	0.064	0.037	0.314	4.55	0.347	1.18	3.76	0.051	0.0056505	0.0152213	171.3	271.3	310.33	12.56
305	0.023	0.039	5.031	0.054	0.035	0.251	4.24	0.287	1.26	4.53	0.046	0.004916	0.0179687	174.3	209	239.33	12.67
335	0.018	0.016	3.722	0.108	0.025	0.205	1.56	1.12	1.32	8.09	0.054	0.007669	0.022555	167.3	183.6	199.33	7.85
370	0.018	0.019	4.377	0.079	0.031	0.225	4.67	0.555	1.14	5.55	0.050	0.0054337	0.0178723	160.6	176.3	206.33	14.53

Table S2. Raw data of chemical elements concentrations and magnetic properties. Weight percentage (Wt. %). Parts per million (ppm). Magnetic susceptibility (MS), low-frequency susceptibility (MS-LF), high-frequency susceptibility (MS-HF), and frequency-dependent magnetic susceptibility (MS-FD).

Table S3. Results of Eigenvalues and Eigenvectors in Principal Component Analysis.										
	PC 1	PC 2	PC 3	PC 4	PC 5	PC 6	PC 7	PC 8	PC 9	PC 10
% variance	43.3	26.03	14.1	7.1	5.2	2.05	0.99	0.62	0.29	0.07
Cum % Var	43.3	69.3	83.4	90.5	95.7	97.7	98.7	99.3	99.6	99.8
Eigenvalue	7.3	4.42	2.41	1.2	0.89	0.34	0.16	0.10	0.05	0.01
Zr	0.28	-0.22	0.22	-0.06	0.05	-0.10	0.48	0.34	-0.06	-0.29
Sr	0.16	-0.02	-0.37	0.08	0.69	0.07	0.13	-0.001	0.45	0.18
Fe	0.30	-0.24	0.11	0.046	-0.002	-0.17	0.01	0.14	0.08	0.0008
Mn	-0.13	0.09	0.33	0.64	-0.15	-0.23	0.13	-0.22	0.44	-0.06
Cr	0.33	-0.07	0.16	0.19	0.01	-0.11	-0.38	0.31	-0.04	-0.12
Ti	0.30	-0.17	0.22	0.04	-0.07	-0.16	0.13	-0.27	-0.03	0.58
Ca	0.25	0.32	-0.04	0.04	-0.21	0.23	0.08	-0.08	-0.14	-0.12
K	-0.33	0.03	0.23	-0.09	-0.16	0.15	-0.06	0.24	0.08	0.53
Al	0.07	0.03	0.52	0.09	0.51	0.25	-0.37	-0.08	-0.28	0.06
Si	-0.35	-0.08	-0.02	0.001	0.08	0.08	-0.16	0.07	0.20	-0.14
Cl	0.02	-0.39	0.22	-0.23	-0.05	0.52	0.28	-0.37	0.13	-0.09
Rb	-0.23	-0.20	0.32	-0.33	0.03	-0.14	-0.06	0.28	0.34	-0.13
Ba	-0.20	0.31	0.15	0.27	0.17	0.26	0.49	0.37	-0.19	0.05
MS	0.16	0.37	0.22	-0.09	-0.004	0.20	-0.12	-0.20	0.31	-0.31
MS-HF	0.18	0.36	0.09	-0.31	-0.01	-0.10	0.05	0.07	0.18	0.14
MS-LF	0.19	0.35	0.08	-0.30	-0.03	-0.07	0.04	0.10	0.20	0.14
MS-FD	0.26	-0.15	-0.18	0.24	-0.31	0.52	-0.19	0.36	0.27	0.13

Table S3. Results of Eigenvalues and Eigenvectors in Principal Component Analysis. Top section presents the Eigenvalues, percentage variation (%Variation), and cumulative percentage (Cum % Var). The bottom section displays the Eigenvectors for variables (chemical elements or magnetic properties).

Magnetic susceptibility

The magnetic susceptibility (*MS*) measurements were analyzed with susceptometer MS2 Bartington instrument, equipped with MS2E frequency sensor of 2 kHz, and MS2B dual frequency sensor with frequencies of 0.465 and 4.65 kHz, with 1% of calibration accuracy (Dearing et al., 1996). All results are expressed in International System of Units (SI). To complement the magnetic properties, we obtain the low-frequency susceptibility (*MS-LF*) and high-frequency susceptibility (*MS-HF*), to calculate the percentage of fine and ultra-fine

grains corresponding to the concentration of superparamagnetic (SP) ferrimagnetic minerals, or frequency-dependent magnetic susceptibility ($MS-FD$), corresponding to $MS-FD$ (%) = $[(MS-LF - MS-HF) / (MS-LF) \times 100]$ (Hunt et al., 1995; Dearing et al., 1996). Also, these magnetic values were normalized to assess their correlation with geochemical data.

4. Results

The results of the dating samples are presented in Table S4. Four of the five values correspond to bioapatite from a Pleistocene horse molar (*Equus* sp.), while the remaining value corresponds to the extinct camelid *Hemiauchenia macrocephala*. Sample UGAMS#73926 is considered invalid due to its age and because it is a specimen recovered from the sieve. Of the remaining samples recovered *in-situ*, sample UGAMS#73925 shows an inverse age, and it cannot be ruled out that this result is due to weathering. In summary, the ages fall within a range of 13,590 to 19,090 $\pm$ 60 BP (see Table S4). The approximate nature of the obtained ages reflects the challenging preservation conditions typically found in tropical environments. Consistent with this, twelve non-diagnostic bone fragments lacking taxonomic features were demineralised for ZooMS and carbon/nitrogen (C/N) analyses. However, poor C/N ratios and the general absence of collagen in most samples ultimately prevented reliable stable isotope measurements for $\delta^{15}\text{N}$, rendering the ZooMS analysis unsuccessful. These samples were excluded from Table S1. In contrast to this diagenetic degradation, the dataset presented in Table S5 deviates from the expected preservation of stable oxygen ($\delta^{18}\text{O}$) and carbon ($\delta^{13}\text{C}$) isotopic compositions in enamel, which typically resists degradation.

Table S4. Radiocarbon dates and associated $\delta^{13}\text{C}$ values from the 'EL Toro' rock shelter at the Loltún cave.														
Sample number	Lab number	Sample	Taxa	Anatomical element	Layer	Depth below datum (m)	Radiocarbon age (^{14}C yr BP)	Calibrated Range (1 σ) (cal BP $\pm$ 1 σ)	a	Calibrated Range (2 σ) (cal BP $\pm$ 2 σ)	a	Median Probability (cal BP yr)	$\delta^{13}\text{C}$ VPDB	Source
LTN-6	UGAMS#73924	Bioapatite	<i>Equus</i> sp.	Molar	El Toro VI/VII	1.6 - 2	18870 $\pm$ 50	22702 - 22940	1	22576 - 22969	1	22802	-3.86	This publication
LTN-7	UGAMS#73925	Bioapatite	<i>Equus</i> sp.	Molar	El Toro XII	3.1	14050 $\pm$ 40	17009 - 17128	0.8	16992 - 17320	1	17087	-3.03	This publication
								17205 - 17241	0.1					
LTN-8	UGAMS#73926	Bioapatite	<i>Hemiauchenia macrocephala</i>	Molar	El toro XIII	2.6 - 3.3	240 $\pm$ 35	0 - 7	0	0 - 24		284*	-10.49	This publication
								152 - 172	0.2	144 - 219	0.3			
								178 - 184	0	264 - 321	0.4			
								278 - 314	0.5	377 - 427	0.1			
								414 - 419	0					
LTN-9	UGAMS#73927	Bioapatite	<i>Equus</i> sp.	Molar	El Toro VI	1.7	13590 $\pm$ 40	16323 - 16481	1	16265 - 16568	1	16407	-3.21	This publication
LTN-10	UGAMS#73928	Bioapatite	<i>Equus</i> sp.	Molar	El Toro VII	1.9	19090 $\pm$ 60	22951 - 23044	1	22909 - 23119	1	23003	-2.82	This publication
	I-11, 103	No. 16: Charcoal	Not identified		-		220 $\pm$ 75	440-0						MARI
	I-11, 104	No. 1: Charcoal	Not identified		-		275 $\pm$ 95	510-0						MARI
	I-11, 036	T-15/17: Charcoal	Not identified		-		945 $\pm$ 135	1130-660						MARI
	I-11, 035	T-12: Charcoal	Not identified		-		1710 $\pm$ 80	1750-1400						MARI
	UCLA-2195A	T-2: Charcoal	Not identified		El Toro V (1977-1980)		2500 $\pm$ 100	2760-2350						MARI
	UCLA-2195B	T-3: Charcoal	Not identified		El Toro V (1977-1980)		1970 $\pm$ 100	2150-1700						MARI
	UCLA-2195C	T-4: Charcoal	Not identified		El Toro VI (1977-1980)		2180 $\pm$ 100	2360-1920						MARI
Unpublished files from Centro INAH Sureste and Middle American Research Institute (MARI) of Tulane University. Housed in the Centro INAH Yucatán.														
a) Relative area under probability distribution														
*This radiocarbon determination is treated as an anomalous result due to significant uncertainty. It is likely to reflect post-depositional disturbance or diagenesis and is therefore not used in subsequent interpretations.														
Calibration to calendar years was performed using the IntCal20 curve and the Calib 8.2 Radiocarbon calibration program.														

Table S5. Stable carbon ($\delta^{13}\text{C}$) and oxygen ($\delta^{18}\text{O}$) isotope values (‰).

Sample number	$\delta^{13}\text{C}$ VPDB	Std. dev.	$\delta^{18}\text{O}$ VPDB	Std. dev.	Taxa	Material	Layer
BULK SAMPLING							
LTN-1	-4.3	0.2	0.7	0.2	<i>Equus</i> sp.	Enamel	VI-VII
LTN-2	-7.9	0.2	-3.6	0.1	<i>Hemauchenia macrocephala</i>	Enamel	IX-XIII
LTN-3	-3.1	0.2	1.5	0.1	<i>Equus</i> sp.	Enamel	VI
LTN-4	-5.9	0.2	-3	0.1	<i>Equus</i> sp.	Enamel	XII
SEQUENTIAL SAMPLING							
Sample Name	$\delta^{13}\text{C}$ VPDB	Std. dev.	$\delta^{18}\text{O}$ VPDB	Std. dev.	Taxa	Material	Layer
LTN-5-1	-4.5	0.3	-1.8	0.1	<i>Equus</i> sp.	Enamel	XII
LTN-5-2	-1.9	0.3	0.8	0.3	<i>Equus</i> sp.	Enamel	XII
LTN-5-3	-2.6	0.3	-0.4	0.2	<i>Equus</i> sp.	Enamel	XII
LTN-5-4	-2.2	0.2	0.5	0.2	<i>Equus</i> sp.	Enamel	XII
LTN-5-5	-3.1	0.3	0.4	0.2	<i>Equus</i> sp.	Enamel	XII
LTN-5-6	-3.4	0.2	0.8	0.1	<i>Equus</i> sp.	Enamel	XII
LTN-5-7	-3.8	0.2	0.5	0.1	<i>Equus</i> sp.	Enamel	XII
LTN-5-8	-3.5	0.3	0	0.2	<i>Equus</i> sp.	Enamel	XII
LTN-5-9	-2.6	0.2	1.7	0.1	<i>Equus</i> sp.	Enamel	XII
LTN-5-10	-3.0	0.2	0.2	0.1	<i>Equus</i> sp.	Enamel	XII
LTN-5-11	-3.3	0.2	0	0.2	<i>Equus</i> sp.	Enamel	XII
LTN-5-12	-2.3	0.30	1.5	0.30	<i>Equus</i> sp.	Enamel	XII



Figure S7. Ceramic shards recovered from the excavation at ‘El Toro’.

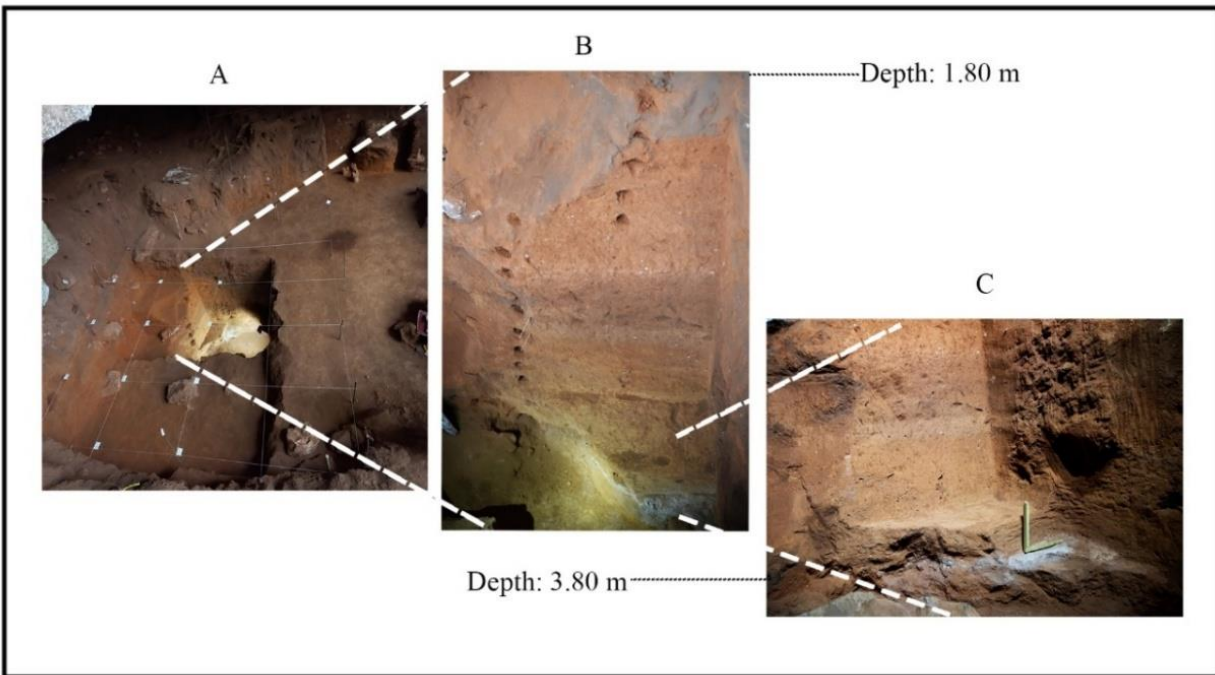


Figure S8. Excavation details from 'El Toro'. A) The georeferenced grid over the excavation pit, B) The west-facing stratigraphic profile of square F13. The continuous vertical sequence indicates the sampling points at 10 cm intervals, C) A detailed view of the deepest level, highlighting the ash deposit (Layer XIV).



Figure S9. Eroded surface associated with Preclassic Maya activity in square G15, the picture shows a stone alignment documented in the northern profile and interpreted as a prepared floor at the base of the ceramic-bearing stratigraphic section.



Figure S10: A Pleistocene horse molar (*Equus* sp.) recovered in situ from square G15, located a few centimetres below the stone alignment interpreted as a ceramic occupation floor. This specimen was sampled for stable carbon ($\delta^{13}\text{C}$) and oxygen ($\delta^{18}\text{O}$) isotope analyses (LTN-3, MPI-GEA_LTN-004) as well as for radiocarbon dating (LTN-9, UGAMS#73927). The results are presented in Tables 1 and S1.



Figure S11. Sample LTN-8 (UGAMS#73926), a camelid (*Hemauchenia macrocephala*) molar, is deemed non-viable and excluded from analysis. This determination is based on inconsistencies in its provenance and apparent age, combined with a significant statistical error in its radiocarbon date.

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