



# Book of abstracts

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**XI-1. Toward the improvement of  
chronological tools to date Lower  
and Middle Palaeolithic settlements  
in Eurasia**

# The late Pliocene prehistoric site of Masol (Northwestern India): new dating of the geological and paleontological context of cut marks and stone tools

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The paleontological site Masol is located in the Himalayan foothills, north of Punjab (India), in the Chandigarh hills. The monsoon combined with tectonics have dug a 60-hectare inlier around a culmination of an anticline. Seasonal torrents release layers of silts and sands within an area rich in mammal and reptile fossils attributed to the late Pliocene (Sahni and Khan 1964, Badam 1973, Nanda 2013). Sahni and Khan have defined the Plio-Pleistocene boundary on sedimentary criteria (pebbles in sands). The torrents are drained by the rivulet Patiali Rao which flows into the Punjab plains. The magnetostratigraphy of its banks locates the fossiliferous lay-

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ers below the Gauss-Matuyama Reversal ( $> 2.58$  Ma) (Ranga Rao 1993). The correspondence between the inversion and the pebbly sands had to be checked. Since 2009 our team surveys the fossiliferous layers ( $n = 1500$  fossils) and collected herbivore bones with cut and percussion marks made by sharp edges in quartzite (Palevol, 2016, 15: 279-452). Their spatial organisation and their trajectories in the sectors of ligament and tendon attachments, enable to reconstitute the manual gestures. Quartzite pebbles have been observed for the first time inside the fossiliferous silts. In 12 localities, fossils have been collected with lithic artefacts (choppers, flakes, hammers, anvils, cores) on surface in permanent erosion. The lithostratigraphic study records 170 meters of sedimentary deposits from the bed of the rivulet Patiali Rao up to the crest that corresponds to the suspected Plio-Pleistocene limit. The fossiliferous layers of 50 meter thickness are located in the lower part of the sequence. The new geological and faunal studies (lithology, mineralogy, granulometry, taxonomy, taphonomy) attest to the fluvio-marshy environments formed by Himalayan rivers flowing into the paleoplains. The layers have restored terrestrial and aquatic vertebrates (e.g. *Merycopotamus*, *Sivatherium*, *Bos*, *Hexaprotodon*, *Testudo atlas*) along with assemblages typical of the Late Pliocene (*Stegodon-Elephas* and *Hipparion-Equus*) and a weak representation of predators (crocodiles, hyenas, *Panthera*). New magnetostratigraphic analyses were firstly performed at the locality where the oldest butchery activities had taken place, close to where a chopper was extracted in stratigraphy for the first time in 2017, and secondly at the highest sequence on the crest. The polarity is normal, invalidating the suspected Plio-Pleistocene lithological boundary (Chapon Sao et al., in preparation). The geomagnetic reversal is pushed higher in the stratigraphy beyond the crest. The pebbly sands are being dated by Electron Spin Resonance (UMR 7194) and by cosmonucleids (at CEREGE by GEOPS). The cut marks and the first chopper *in situ* at Masol 1 are 140 meters below these sands, the result of the dating will be presented and the implications discussed.

**Keywords:** Masol, prehistory, India, late Pliocene, dating, cosmonucleids, ESR

# Molluscan Pleistocene biozonation : dating Acheulean occupations in Europe

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Recent malacological investigations at several classic sites from Northern France and central Italy together with reappraisal of old palaeontological collections provide new informations leading to palaeoenvironmental reconstructions contemporaneous with Acheulean settlements. Molluscan biozonation defined from these studies are reinforced at several locations by physical dating methods (U/Th, ESR, TL, 40Ar/39Ar). In France, the studied sites are located in the Somme and the Seine basins and within this area Acheulean occupations span from the Late Cromerian (MIS 15) up to the Late Middle Pleistocene (MIS 9). In Italy, recent investigations were undertaken in the Abruzzo and highlighted malacological successions associated with several Acheulean settlements from MIS 14 up to MIS 12.

This contribution presents a synthesis of the European malacological data available and review both quality and accuracy of the information.

Some phases, like the MIS 11 interglacial appear particularly well documented with numerous sites in north-west Europe including good malacological record allowing recognition of particular species, i.e. presence of critical forest species, especially those now extinct or occurring far beyond their modern ranges. They also give way to detailed landscape reconstructions. In such case environmental successions defined from malacological series provide a framework of the temperate episode within which associated Acheulean industries can be set. These results allow to precise the chronological prehistoric settlements succession that is out of reach of physical dating technics. In the Mediterranean area malacological references are still under construction but recent results give way to the first step of a regional framework and allow comparison of the malacological responses to climatic cyclicity between northern and southern Europe.

**Keywords:** Malacology, Middle Pleistocene, Europe, Chronology

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# New chronological data at South African Middle Stone Age sites applying femtosecond laser ablation – ICPMS for u-series dating of marine shells, snail shells, ostrich eggshells and tooth enamels.

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The dating of Middle Stone Age sites in South Africa has been mainly performed with luminescence dating methods on quartz or feldspars grains from sediment and burnt lithics. In comparison, U-series dating of biominerals has been less exploited. This is due to the difficulty 1) to overcome or model the eventual late input of uranium and thorium in the material and 2) to measure the low U and Th content in the materials where this input did not occur. Here, we present results taking advantage of a high repetition rate femtosecond laser-ablation system coupled with a HR-ICPMS from the Pau University that allows overcoming these limitations. Indeed: high resolution maps of U and Th contents can be obtained within a reasonable time length and the very high sensitivity achieved by this equipment allows measuring concentrations as low as a few ppb and ppq of material. Ostrich eggshells, giant snail shells, marine shells and tooth enamel from Bushman Rock Shelter and Sibudu Cave have been studied.

**Keywords:** U, series dating, Middle Stone Age, ICPMS, femtosecond laser, biominerals

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# On the limit of U-uptake modelling for ESR/U-Th dating of teeth with leaching issues: the case of Sainte-Anne I site

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The reconstruction of the unknown U-uptake history in the dental tissues has always been a source of uncertainty for ESR/U-Th dating and thus for dose rates reconstruction. Since the introduction of the US-ESR model by Grun et al. (1988), which takes into account  $^{234}\text{U}/^{238}\text{U}$  and  $^{230}\text{Th}/^{234}\text{U}$  activity ratios for the determination of an uptake parameter (the "p-value"), the accuracy of the age estimates was improved. However, the limit of the US-ESR model is reached when uranium leaching is evidenced in at least one of the dental tissues. In order to overcome this limit, Shao et al. (2012) proposed a new model, called Accelerating Uptake (AU-ESR), which allows to model the evolution of U-uptake as a function of time in tissues which underwent leaching processes.

We present ESR/U-Th age results obtained on *Equus* teeth from the Middle Palaeolithic cave site of Sainte-Anne I (Polignac, Haute-Loire, France) (Raynal, 2007). Results suggest that the dental tissues from five out of six teeth underwent U-leaching during burial, preventing the calculation of US-ESR ages. Minimum ages (Early Uptake, EU-ESR) were thus calculated and compared to AU-ESR modelled ages. The difference between mean EU and AU ages ranges from 2 to 22%, indicating that U-loss in some of the dental tissues can have a significant impact on the age estimates. Available biochronological and climatochronological data point out a MIS6 chronology. However, previous ESR ages (Rhodes, 2007) and all of the EU, US and AU ages

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obtained in this study fall within MIS5. ESR/U-Th ages range from  $118 \pm 14$  ka (AU) to  $80 \pm 7$  ka (US), spanning the entirety of MIS5, and allow to attribute the sequence to the beginning of the Upper Pleistocene.

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**Keywords:** Chronology, ESR/UTh, Neanderthal, Middle Palaeolithic, Western Europe

# Extended-range luminescence dating of fluvial terraces (Duero basin) associated with La Maya I, II and Burganes Lower Palaeolithic (Acheulean) sites, west-central Spain

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A large proportion of the Lower Palaeolithic (Acheulean) records from the Iberian Peninsula are associated with fluvial archives located in the major river basins of the Duero, Tajo and Guadiana drainage systems. Unfortunately, these open-air archaeological records are often difficult to constrain chronologically due to a lack of suitable material for dating (e.g., faunal remains) or because their Middle Pleistocene antiquity precludes the use of otherwise applicable geochronometric techniques, such as optically stimulated luminescence (OSL) dating of sedimentary silicates. As a result, many important Acheulean sites lack precise and accurate chronologies, and their existing temporal frameworks are based solely on morphostratigraphic correlations with regional fluvial terrace systems. Over the past 10 years, a series of ‘extended-range’ OSL methodologies have been proposed for dating Middle Pleistocene deposits, which make use of luminescence signals with higher dose saturation limits. These techniques include post infrared - infrared stimulated luminescence (post IR-IRSL) dating of K-feldspars and thermally transferred-OSL (TT-OSL) dating of quartz.

This study reports on the application of these extended-range luminescence methods to Lower Palaeolithic (Acheulean) sites from central-west Spain. We present results of an ongoing dating program that aims to determine the chronologies of a number of fluvial terraces associated with the archaeological sites at La Maya I, II and Burganes, all of which have produced important lithic assemblages.

The sites of La Maya I, II and Burganes are located in the western sector of the northern Meseta (Spain), within the Duero drainage system. The La Maya I (+6-8 and +14 m above river level) and II (+34 m) sites and the Burganes sites (+16-20 m and +34 m) are situated on the middle terraces of the Tormes and Tera rivers, respectively. In total, seven optical dating samples were collected across the various river terraces: one sample was collected from La Maya I (+6-8 m) and one sample was collected from La Maya II (+34 m); two additional samples were obtained from the main excavation site of La Maya I (+14 m); two samples were taken from the

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+16-20 m Burganes terrace and one sample was collected from the +34 m Burganes terrace. Extended-range luminescence ages were determined using multi-grain post IR-IRSL dating of aliquots of K-feldspar grains and single-grain TT-OSL measurements made on individual silt-sized quartz fractions. Ages were calculated using dose rate values obtained from a combination of *in situ* gamma spectrometry and beta counting. We report on the suitability of the post-IR IRSL and TT-OSL signals for dating these deposits, which has been tested via a series of quality assurance criteria (signal composition, dose recovery tests, bleaching tests, sensitivity correction assessments). The resultant chronologies are presented and used to assess (i) the accuracy of existing chronostratigraphic frameworks developed at these sites, and (ii) the wider applicability of these novel dating approaches for constraining open-air, Lower Palaeolithic chronologies in the Iberian peninsula.

**Keywords:** Luminescence dating, Lower Palaeolithic, Acheulean, La Maya, Burganes, Spain, fluvial terraces

# New methodology using femtosecond laser ablation ICP-MS for direct U-series disequilibrium dating of ostrich eggshell fragments

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During the Middle Stone Age in South Africa, anatomically modern humans developed complex behaviors involving symbols and art. Huge efforts have been done for establishing the chronology of the emergence and spreading of these behaviors. However, the time range is over the limits of the C14 dating, and the complexity of the sedimentary deposits results in methodological issues for luminescence dating. In order to enrich the chronological tools, we have adapted the U-series disequilibrium dating method applied to ostrich eggshell fragments: a new protocol for direct isotopic measurement with femtosecond laser ablation ICP-MS has been developed. This method only necessitates a few milligrams of matter, making it usable on very small fragments. Moreover, it does not require heavy chemical preparation and is efficient even for low concentration (ppb) of uranium. Therefore this method opens new possibilities for absolute dating and chronological studies. Here, we present our protocol, as well as noticeable considerations on sample conservation and uranium uptake and leaching phenomena.

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**Keywords:** dating, ostrish eggshell, U series disequilibrium, laser ablation, ICP MS

# Direct ESR dating of *Homo antecessor* from Atapuerca Gran Dolina TD-6 (Spain): first results

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We present here the details of the first direct Electron Spin Resonance (ESR) dating study of *Homo antecessor*, the earliest known hominin species identified in Europe. The analysis of a tooth (ATD6-92) from TD6 unit of Atapuerca Gran Dolina (Spain) was carried out following a "semi non-destructive" procedure combining ESR measurements of an enamel fragment and high resolution Laser Ablation ICP-MS U-series of dental tissues. Additional magnetostratigraphic data were collected within TD6 for independent age control. Our dating results are consistent with previous studies of TD6 unit and associated fossil remains and confirm the Early Pleistocene age of *H. antecessor*. Additionally, this work illustrates the challenge of dating fossil human teeth by means of ESR. It identifies the specific pitfalls inherent to this application, in particular the systematic  $\mu$ CT-scanning of human remains, or the existing uncertainty arising from the absence of the original surrounding environment, which complicates the dose rate reconstruction. Other sources of uncertainty are common to standard ESR dating applications to large mammal fossil teeth, such as the spatial distribution uranium-series isotopes in dental tissues, the variability of the water content over time, or the potential preferential creation of unstable NOCORs in the radiation-induced ESR signal. Pre-screening of fossil remains using laser ablation IPC-MS appears to be essential prior to any subsequent analysis to evaluate the suitability of the sample for combined US-ESR dating, although it does not preclude future complications in the dating process.

**Keywords:** Quaternary Geochronology, Lower Palaeolithic, ESR dating, Atapuerca, *Homo antecessor*

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# The chronological framework of the Italian Peninsula Early-Middle Pleistocene sites : recent advances and what's next ?

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The attempts to reconstruct migration paths, understand techno-cultural evolution or place archaeological sites within existing palaeoclimatic and palaeoenvironmental reconstructions during the Middle-Pleistocene are hampered by the lack of a unified and precise chronological framework. In order to unlock this scientific barrier, an intense effort to improve the accuracy and create a unified chronological framework from the central and southern Italian peninsula was undertaken in the frame an Italian-French 6 years collaboration including geochronologists, geologists and prehistorians from various institutions. For the first time in Western Europe the  $40\text{Ar}/39\text{Ar}$  method combined with palaeo-dosimetric methods was systematically used in sites ranging in age from 700 to 300 ka. This geochronological work was made in parallel to the improvement of the regional Middle-Pleistocene tephrostratigraphic database thanks to new high-precision  $40\text{Ar}/39\text{Ar}$  dates as well as new geochemical works. The chronological framework we build is constantly improved by new data and currently allows replacing 20 archaeological and palaeontological sites into their regional and global climatic and environmental contexts. Our work provides to archaeologists the opportunity to compare lithic industries from technical and evolutionary points of view within a homogeneous temporal reference at the local and regional scale as well as to propose a numerical age for the Galerian and Aurelian faunal units limit. The approach developed is unfortunately restricted to the Italian peninsula so far. However, the rapid technical and methodological improvements of the  $40\text{Ar}/39\text{Ar}$  method as well as of the European tephrostratigraphical database (i.e. chemical and isotopic fingerprinting) will allow in the near future the study of more distal volcanic material.

**Keywords:** Geochronology,  $40\text{Ar}/39\text{Ar}$ , Middle Pleistocene, Italian peninsula

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# ESR-U-series dating of Caune de l’Arago: discussion between sediment characteristics and dose rate calculation in a dense and diversely weathered Middle Pleistocene archaeological layer

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Caune de l’Arago is a karstic cave located between the Mediterranean Sea and the Pyrenees. Excavations have revealed thirteen meters of its infilling, in which 55 Middle Pleistocene occupation layers have been individualized through the study and distribution of hundreds of thousands of faunal and lithic remains. The correlation to the LR05 Marine Isotopic Stage (MIS) curve, based on palaeoenvironmental, radiometric and biochronological data, attributes the excavated layers to MIS 15 to 5 and thus place them among the oldest Acheulean layers in Europe and among the rare sites with human remains of this age.

However, the richness and density of these anthropogenic occupation layers, their inhomogeneous degree of weathering, make the radiometric age, and more specifically the annual dose, difficult to calculate.

The ESR-U-series dating of the G archaeological layers have led to diverse ages. Three groups can be individualized, according to the nature of the embedding sediment, and we propose here to evaluate how the different textural, mineralogical and micromorphological data may interfere with the age calculation.

One scenario for the diagenesis of the sediment is that the sampled teeth were deposited in a probably open rubble, more or less filled in during MIS 12 and locally or totally cemented by secondary calcium carbonates from MIS 11 on, or transformed from calcium carbonates to calcium phosphates from MIS 5 on.

Whatever the option we prioritize for interpreting the datings, this site offers the opportunity to quantify the amplitude of changes affecting the age calculation for a single archaeological unit and may be remembered as a good reference for Middle Pleistocene karstic contexts.

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**Keywords:** ESR, U, series dating, Middle Pleistocene, Diagenesis, Dose rate, Caune de l'Arago

# Hominid early migrations into Eurasia: A paleomagnetism-ESR perspective

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The so-called Early Middle Pleistocene Transition (EMPT), from 1.4 to 0.4 Ma, was a major revolution in the Quaternary. For about one million years several events including a progressive increase of climate oscillations, a change in orbital rhythm from 41 to 100 ka cycles, and an increase of the long term ice volume, led to dramatic changes in the biota including a major faunal turnover and hominid dispersal and speciation. An increase in Saharan terrigenous dust flux as observed in both the Mediterranean Sea and Arabian Sea, further supports the beginning and the role of the EMPT. Overall, the existing data reveal that the arrival of hominids to Europe was apparently favored by periods of extreme climate variability, conditions that are met as early as 1.4 Ma. Two major geomagnetic chrons are critical to identifying the EMPT interval in sedimentary sequences, including the Matuyama-Brunhes Boundary (0.78 Ma) and the Jaramillo Subchron (0.99-1.07 Ma), with the latter placed in the middle of the EMPT. Therefore, the Jaramillo is a key time marker that allows placing fossils and archaeological remains within the Early Pleistocene and EMPT. A number of European localities with important faunal remains have become "classic" references as the Jaramillo Subchron has been purportedly identified. We have re-evaluated such localities in order to assess the assumed chronology of the fossiliferous and/or archaeological record. Issues related to rockmagnetism, data quality, and remagnetization among other, make us to consider some of these sites with caution. Nevertheless, when combined with numerical dating methods such as Electron Spin Resonance, magnetic reversal stratigraphy has been proven to be a powerful tool to assign and to constrain chronology to sedimentary sequences that formed during the EMPT thanks to the presence of the Jaramillo Subchron.

**Keywords:** Eurasia, Lower Paleolithic, Pleistocene, geochronology, paleomagnetism, ESR

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