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# Journal of Archaeological Science: Reports

## First AMS $^{14}\text{C}$ dates on the Protoaurignacian in Mediterranean France: the site of Esquicho-Grapaou (Russan-Ste-Anastasie, Gard)

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<b>Abstract:</b>	<p>This paper presents the first AMS radiocarbon dates done on the Protoaurignacian layer (SLC1 a+b) of Esquicho-Grapaou, a stratified site in Southeastern France. Previous conventional method radiocarbon dates at this site (mostly on charcoal) done in the 1970s produced too large standard deviations, making them difficult to place precisely in time, but already pointing to the antiquity of this layer. For AMS radiocarbon dating we selected taxon-identified faunal samples of the 1970s Bazile excavation collection. Of six samples attempted, two produced dates. These are the first AMS <math>^{14}\text{C}</math> Protoaurignacian dates in Mediterranean France. In this paper they are placed within a larger context of recently-dated Protoaurignacian sites in western Mediterranean Europe. The Esquicho-Grapaou dates fall squarely in the middle of these, in the 38.7-41.9 ka cal BP range (95.4%), fully in-line with what is currently known about the timing of Protoaurignacian presence in western Mediterranean Europe.</p>
<b>Suggested Reviewers:</b>	<p>Rachel Wood, PhD Australian National University rachel.wood@anu.edu.au Expert in radiocarbon dating, including sites of this time period.</p> <p>François Bon, PhD Universite Toulouse Jean Jaures francois.bon@univ-tlse2.fr Expert on Aurignacian lithic technology in France</p> <p>Jean-Jacques Hublin, PhD Max-Planck-Institut fur evolutionare Anthropologie hublin@eva.mpg.de Expert in human evolution, especially Neanderthals and Homo sapiens and their interaction</p> <p>Steven Kuhn, PhD University of Arizona skuhn@email.arizona.edu Expert in the Aurignacian of the Mediterranean region</p> <p>Tim Jull, PhD University of Arizona jull@u.arizona.edu Expert in AMS radiocarbon dating.</p>
<b>Opposed Reviewers:</b>	

April 30, 2020

Editorial Board, Journal of Archaeological Science: Reports,

Please find attached a manuscript, entitled, “First AMS  $^{14}\text{C}$  dates on the Protoaurignacian in Mediterranean France: the site of Esquicho-Grapaou (Russan-Ste-Anastasie, Gard)” which I and my co-authors would like to have considered for publication in the Journal of Archaeological Science: Reports.

Our ‘Highlights’ summarize what we have done in this research, namely:

Dated the Protoaurignacian layer of Esquicho-Grapaou, France (6 samples, 2 results)

These are the first AMS  $^{14}\text{C}$  dates on the Protoaurignacian in Mediterranean France

Synthesis and new data on biotic context (fauna, charcoal, pollen)

Occupation at Esquicho-Grapaou in middle of Protoaurignacian range of western Europe

Protoaurignacian dates overlap those of last Neanderthals

This article provides the first ever AMS radiocarbon dates for this techno-complex in this region. This region is of importance to the dispersal of anatomically modern *Homo sapiens* across Europe. As you are aware, accurate and precise dating of the early Upper Palaeolithic is critical to our understanding of the possible relationship between final Neanderthals and early modern humans in Europe. As such, a rigorous set of sample selection and evaluation protocols was developed and used in this research, which is described. These results are placed in a larger context of Neanderthal presence, as well as Châtelperronian and Uluzzian dates.

In 2018, the *Journal of Archaeological Science: Reports* published our results from the Aurignacian site of Isturitz (<http://dx.doi.org/10.1016/j.jasrep.2017.09.003>). In that article we also summarized, plotted and discussed dates for Protoaurignacian sites across western Europe. At the time, no Mediterranean France Protoaurignacian sites had been dated successfully. The present article attempts to fill that gap.

I am fully bilingual in English and French. Thus, referee comments may be written in either of these languages, without the need for translation.

Thank you very much, in advance, for your time and effort in considering this manuscript. I look forward to hearing from you.

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## First AMS $^{14}\text{C}$ dates on the Protoaurignacian in Mediterranean France: the site of Esquicho-Grapaou (Russan-Ste-Anastasie, Gard)

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### Abstract

This paper presents the first AMS radiocarbon dates done on the Protoaurignacian layer (SLC1 a+b) of Esquicho-Grapaou, a stratified site in Southeastern France. Previous conventional method radiocarbon dates at this site (mostly on charcoal) done in the 1970s produced too large standard deviations, making them difficult to place precisely in time, but already pointing to the antiquity of this layer. For AMS radiocarbon dating we selected taxon-identified faunal samples of the 1970s Bazile excavation collection. Of six samples attempted, two produced dates. These are the first AMS  $^{14}\text{C}$  Protoaurignacian dates in Mediterranean France. In this paper they are placed within a larger context of recently-dated Protoaurignacian sites in western Mediterranean Europe. The Esquicho-Grapaou dates fall squarely in the middle of these, in the 38.7-41.9 ka cal BP range (95.4%), fully in-line with what is currently known about the timing of Protoaurignacian presence in western Mediterranean Europe.

**Keywords:** Radiocarbon dating; Middle-Upper Palaeolithic transition; Protoaurignacian; Modern humans; Sample selection-methodology; Mediterranean France

### 1. Introduction

Chronometric dating has played a critical part in discussions about the Middle to Upper Palaeolithic transition. Radiocarbon dating in particular has been used to assess the extent of possible contemporaneity of technocomplexes (indirectly linked to species), the antiquity of innovations and even the stratigraphic integrity of sites, among other issues (Hublin et al., 2012; Douka et al., 2014; Wood et al., 2018). Recent advances in laboratory methods, coupled with Bayesian statistical analysis and other modelling methods, have altered the timing of events, often pushing them further back in time and resulting in revised models of species interaction, changes in likely authorship of innovations and alternate views regarding timing of dispersal as well as the role of climate in instigating change

(Higham et al., 2009, 2012; Szmids et al., 2010a; Banks and d'Errico, 2013a, b; Wood et al., 2013; Deviese et al., 2017).

Careful selection of samples to be dated is also critical to the accuracy of the results, but these are not as often emphasized. This includes stratigraphic analysis to ensure selection from least disturbed squares and from the correct layer (e.g. Barshay-Szmids et al., 2012), faunal analysis to select humanly-modified animals (Szmids et al., 2010b; Wood et al., 2014, 2018; Barshay-Szmids et al., 2018b) and dating of identified individual charcoal, originating directly from hearths (Wood et al., 2012; Barshay-Szmids et al., 2018a). These criteria raise the issue of the importance of geomorphological and taphonomical aspects to better control the site-formation processes. By adopting such practices as part of protocol, one can improve the likelihood that the resulting dates reflect human occupation.

The Protoaurignacian (PA), in particular, has received more attention in the last decade, as an early case of Upper Palaeolithic culture. In Southeastern France, in Mediterranean areas, the PA seems to be the earliest cultural manifestation of anatomically modern *Homo sapiens* (AMHS). Although hominin fossil evidence is extremely scarce for this technocomplex, a recent analysis found that two deciduous incisor teeth associated with PA layers (at Riparo Bombrini and Grotta di Fumane, in Italy) belong to modern humans (Benazzi et al., 2015). In addition to the fact that the PA always occurs before other Aurignacian variants such as the Early Aurignacian (EA) when the two are stratified at a site, a recent re-evaluation of PA and EA dates across western Europe showed that the PA begins earlier; however, after the initial manifestations, there seems to be a regional-level chronological overlap of these two early Aurignacian industries (Barshay-Szmids et al., 2018b). There also seems to have been regional variation in PA practices (Falcucci et al., 2017), thus warranting independent regional assessments and comparisons. At the dawn of AMHS dispersal into Western Europe, it is essential to determine the potential extent of species interaction and demise, for which the accuracy and precise dating of the Protoaurignacian in each region has become crucial (Hublin et al., 2015).

For the Middle to Upper Palaeolithic transition in southern France, one of us (C. B.-S.) initiated a regional-scale project to date a number of Aurignacian and Mousterian sites using improved laboratory and field selection techniques (Szmids, 2010 a,b,c; Barshay-Szmids et al., 2012, 2018a,b). One of the sites included in this project was Esquicho-Grapaou (Fig. 1), a cave-site containing an archaeological level which was one of the first to have been recognized (Bazile et al., 1981) as belonging to what was later termed 'Protoaurignacian'

## **2. Brief history of excavation and Site description**

Located just west of the Rhône river, in the Gard river gorges region (*Le Gardon*), Esquicho-Grapaou, which translates as 'crushed toad' in the local Provençal language, is a cave site consisting of fairly narrow corridor-like passages, some of which were excavated early in the 20<sup>th</sup> century (Fig. 2). In the early 1970s, a rescue excavation was made in the deeper part of the cave, totaling 10 m<sup>2</sup>, to prevent further degradation of the *in situ* deposits (Bazile, 1974, 1976).

The stratigraphy is essentially composed of three main archaeological layers (Fig. 3), although, like most cave sites, such layers are not as clear in all zones of the site. Located at the main entrance of the cave we have a relatively cemented cryoclastic stone layer (CC2) bound by carbonates at the top (BR2) containing a Quina Mousterian industry. After a hiatus, or erosive phase, in sedimentation, and located in the deepest part of the cave, we found a weathered and altered level with stones and clay, often colored by red ochre and in which is located the Protoaurignacian (SLC 1a+b). At the top of this

sequence, encrusted angular stone levels (named CC1-BR1) contain a line of hearths (F1) and yield a relatively poor Early Aurignacian (Aurignacian I) assemblage.

Early 70's research and survey was conducted by one of us (F.B) in the Gardon river valley to locate Upper Palaeolithic sites. After excavating what was left of the La Laouza rock-shelter from the Abbé Jean Bayol excavation (and destruction) of the 1930s (Bazile et al., 1981), fieldwork was conducted at Esquicho-Grapaou. The material from these two sites revealed a previously unknown archaic lithic industry in Southeastern France attributed to an early phase of the Aurignacian (Bazile et al., 1981). Although the Esquicho-Grapaou 1970s excavation was not extensive in area, it revealed a wide array of materials including lithics, fauna and colorants. These have been described in previous publications, especially the lithic material (Brugal, 1981a; Bazile and Sicard, 1999; Bazile, 2002, 2004, 2005) so will not be elaborated upon here except as they pertain to the techno-typological attribution of industries and/or link with human occupation, as well as a better description of the faunal associations and paleoenvironmental context, which can be relevant regarding dating. All of the Esquicho-Grapaou material is currently stored at the *Musée National de Préhistoire* (Les Eyzies-de-Tayac, Dordogne).

### **3. Lithic industries, Faunal associations and Palaeoenvironmental context**

From the start it was realized that the lithics from SLC1a+b were unusual compared to what was known at the time (1970s) regarding the Aurignacian, especially compared to the neighbouring sites of La Salpêtrière (Remoulins) and La Balauzière (Vers-Pont du Gard). These differences were fully described (Bazile 1974, 1976 and later 2002, 2005), but overall it was felt that an Aurignacian techno-complex was the most appropriate attribution. Its unusual aspects led Bazile to ascribe the lowermost Aurignacian (SLC1a+b) provisionally to Delporte's '*Aurignacien O*' of southwestern France, possibly contemporary with the *Périgordien ancien* (Châtelperronian), and older than the Classic *Aurignacien I*. The assemblage from CC1-BR1 was considered Aurignacian I (Bazile 1976, 2002).

Research on the Aurignacian in western Mediterranean Europe over the next thirty years, in particular more extensive comparisons between lithic material from Esquicho-Grapaou with sites in Spain and Italy by one of us (F.B.), as well as more recently, detailed technological lithic studies of Mediterranean Aurignacian sites (Sicard, 1994, 1995; Bon, 2002) enabled the diversity of the Aurignacian, *sensu lato*, to be revealed. SLC1a+b are surely attributed to the Protoaurignacian (PA) and BR1 to the Early Aurignacian (EA) (*sensu* Bon, 2002, 2006). The rest of this article will focus on layers SLC1a+b, aiming to date the first moment of Upper Palaeolithic occupation in this region of southwestern Europe.

A number of characteristics of the lithics point to SLC1a+b as very similar to La Laouza rock-shelter (Sicard, 1994, 1995; Bazile and Sicard, 1999; Bazile, 2002, 2005; Bon, 2002) containing a Protoaurignacian industry. This lithic industry includes a continuity between blades and bladelets (one operational scheme producing both, as opposed to separate *chaînes opératoires* for each), rare carinated/muzzled endscrapers, very rare Aurignacian retouch on blades, a dominance of endscrapers over burins, a high proportion (60%) of large (40 mm+ in length) rectilinear Dufour bladelets, as well as smaller rectilinear Dufour bladelets (20-30 mm in length) (Figs. 4 and 5). The Esquicho-Grapaou lithic assemblage represents a total of 1800 artifacts with at least 120 retouched pieces (Table 1). The red colorants have been sourced to a nearby location, less than 5-10 km, from old karstic infilling of the Urgonian plateau on the left bank of the Gardon. One sagaie made from cervid antler and one ivory ornamental pendeloque (Fig. 6) broken at the suspension hole and very likely coated in red ochre (length of object is 20 mm) complete the set. We suspect the presence of shells, although not preserved here, given that they have been found in the La Laouza site.

The bone remains from CC2 – NISP= 300: excavations C. Hughes and S. Gagnière (1931) then F. Bazile – are very well preserved. The faunal association is mostly composed of reindeer (64%), horse (24.3%) and large bovid (cf. *Bison*, 5.3%) with few ibex and wolf (2% each), deer and giant deer (1% each) and lynx (0.3%), all of which represent a typical cold association. The bone elements display much evidence of anthropic activities (cut-marks, notches,...) and few carnivore marks; some predator pits overlap human cut-marks. The fauna from SLC1 (Table 2) is different and globally well-preserved (weathering stage mostly stage 0) but more fossilized (phosphate and manganese) and showing sedimentary crust, limited concretions and dry breakage. The association consists primarily of horse (as in BR1) followed by ibex and large bovid, with few remains of *E. hydruntinus* and small-sized deer (cf. *Capreolus*). If we only consider herbivores, equids represent ca. 75%, ibex ca. 9% and bovine ca. 6%. All age classes are present for horses, and adults are dominant in this assemblage. The carnivores are scarce with cave hyena (only represented by one coprolite), wolf and a small mustelid. The absence of reindeer remains as well as the presence of hydruntine could indicate more temperate or cooler climatic conditions for this level, suggesting a relatively open environment. It is interesting to note that the faunal association from the Protoaurignacian of La Laouza (level 2b1) is similar to the one from Esquicho-Grapaou with horses – both cabaline and hydruntine – ibex and bovine, and cave hyena (Brugal, 1977, 1981b).

The bone assemblage is highly fragmented and not very abundant in determinable remains, but bone splinters can be assigned to size-classes and attributes (i.e., presence of spongy parts on the internal surface of bone shaft, cortical thickness) are mostly indicative of equid taxa. Human activities on bones are essentially present on horse and ibex remains, with cut-marks, chop-marks and a few bone flakes (see Figs. 7 and 8); green fractures on fresh bone are relatively frequent and not associated with carnivore marks, the latter being uncommon. The presence of burnt material, mainly represented by small fragments, is rather important (use of bones as fuel?). On the whole, most of the bone assemblage of SLC1 a+b results clearly from anthropic subsistence activities. The dated samples were selected among the human-modified material.

The ten minimal ungulate individuals of different size (from large bovid to roe deer) found at Esquicho-Grapaou suggest a generalist acquisition and hunting strategy, similar to the one described recently in the Protoaurignacian levels of Riparo Bombrini (Italy). The two PA levels of this Italian site are respectively interpreted as residential mobility in a temperate climate (A1) followed by logistical mobility (A2) strategies under colder climate (Bouchard et al., in press).

All the levels of Esquicho-Grapaou yielded charcoals which were analyzed by Bazile-Robert (1979, 1981). The CC2-BR2 levels (n=34 charcoals) show a vegetation dominated by Scots pine (*Pinus sylvestris*, 55%), willow and poplar, indicating a cold and dry climate. The SLC1b level (n=216) has a similar vegetation with Scots pine (85%), willow/poplar (6.8%) and rare birch (1.1%). The Scots pine (44%) is still dominant in SLC1a (n=88) associated with juniper (10.6%) and diverse deciduous trees (*Betula* sp., *Hippophae rhamnoides*, *Salix* sp., *Fagus sylvatica*) and several rosaceae (*Prunus mahaleb*, *Sorbus domestica*, *Crataegus monogyna* and *Viburnum lantana*). The SLC1 levels show a wide biodiversity, markedly forested or shrubby and reveal a climate more temperate and humid than the previous one, probably related to climatic instability. This meets the anthracological results obtained at La Laouza, which includes Scots pine (80%) and some Mediterranean taxa, such as sclerophyll oak *Qercus ilex-coccifera* (8%) and sea buckthorn *H. rhamnoides* (1.6%). Finally, the CC1/BR1 levels (n=93) indicate an even more open vegetation under colder and less wet climatic conditions, primarily showing willow (*Salix* sp. 46%), Scots pine (23.6%) and juniper (8.6%), with very few leafy taxa and an absence of Mediterranean taxa.

Thirteen palynological samples through the sequence (Farbos, 1984; see Fig. 3), of which only five had no data, bolster the charcoal analysis. They indicate the constant presence of Scots pine and other trees (*Betula*, *Tilia* and *Cupressaceae*) and a dominance of gramineae and heliophil herbaceous plants (*Crucifera*, *Chenopodiaceae*, *Rubiaceae*...). Overall, the environment shows a low arboreal canopy developed during moderate (SLC1) or colder conditions (CC2-BR2 and BR1), with more humidity in SLC1. It is interesting to note that both among charcoal and pollens, five taxa have a food interest in that they bear edible raw fruit (sea buckthorn, cormier, monogyny hawthorn, hazel and one rosacea). Finally, in agreement with the faunal data, the Esquicho-Grapaou sequence demonstrates a climatic fluctuation, with a temperate phase (SLC1, formerly attributed to the end of interstadial Würm II-III as a catathermic period) framed by two colder periods.

#### **4. Previous dating**

In the mid-1970s, bulk charcoal samples from each of the Aurignacian layers (BR1 and SLC1a+b) were sent to the Monaco Radiocarbon Laboratory for conventional radiocarbon dating, producing one date for BR1 and a few dates for SLC1a+b (Bazile, 1976, 1979). The large standard deviations, however, made them difficult to attribute precisely. Bones from the Mousterian layer were sent to the Lyon Laboratory, but produced a date far too young for such an industry and not in accordance with stratigraphy (see Table 3) (Delibrias and Évin, 1980: 220; Évin et al., 1983: 119)

#### **5. New radiocarbon dating**

As part of a larger southern France radiocarbon dating project (see above), we selected six Esquicho-Grapaou samples from the 1970s F. Bazile excavation to submit for AMS radiocarbon dating. We also selected six bones from the neighboring sites of La Laouza Layer 2b1 (Protoaurignacian) and two from La Salpêtrière (Early Aurignacian), also excavated by F. Bazile in the 1970s and often considered together in publications. Bones from the collections were selected by the three of us together and one (J.-P. B.) examined them taxonomically and taphonomically to identify them as far as possible. For the three sites we sampled bones of good length (our range is 4 to 12.7 cm), to minimize possible vertical movement of samples in the stratigraphy, and of large mass to maximize collagen preservation potential. We chose samples that could be identified as precisely as feasible and gave preference to those displaying human modification (see above).

For Esquicho-Grapaou, we focused especially on bones from band T, where SLC1a+b (PA) is clearly separated from the underlying Mousterian (CC2-BR2) layer and from the overlying Early Aurignacian (BR1) layer. Five bones from SLC1a+b were submitted for AMS radiocarbon dating to the Oxford Radiocarbon Accelerator Unit (ORAU, Oxford, England). All have spiral fractures (i.e. green fractures). Another had been submitted a few years earlier, to the now-defunct IsoTrace AMS Laboratory (Toronto, Canada). Complete sample information is presented in Table 4.

#### **6. Laboratory methods**

##### **6.1 IsoTrace Laboratory**

The sample done at IsoTrace was processed according to the following protocol. The surface of the bone was physically cleaned, then a piece of compact bone was cut off and crushed to a fine powder. This powder was divided into three batches, with each being chemically pretreated and having collagen extracted, as described below. The batch with the highest collagen percentage yield was the one subjected to further processing. Pretreatment and collagen followed a modified Longin method (1971).

Each batch of powdered bone was demineralized in cold 1.0 N HCl (4-6 °C) to minimize collagen loss. The residue was desalted and washed to neutrality to remove any acid soluble contaminants. Cold NaOH was then used to wash out any alkaline soluble contaminants. The raw collagen was then gelatinized in hot acidified water to remove potential acid and alkali insoluble contaminants. IsoTrace used a refrigerated high-speed ultracentrifuge at 25 000 *g* to separate supernatants from insoluble residues. This high *g* force allowed for improved separation of supernatants from insoluble residues and contaminants compared with a regular centrifuge. The purified collagen weight and yield were calculated after being lyophilized (freeze-dried). In the case of Esquicho-Grapaou, collagen yields were extremely low for all batches. Some batches were combusted but yields were far too low for further analysis.

## 6.2 Oxford Radiocarbon Accelerator Unit

Bones submitted to the ORAU Laboratory were processed according to their standard protocols for bone. These are published (Bronk Ramsey et al., 2002, 2004a,b; Higham et al., 2006; Brock et al., 2007, 2010), but summarized below. The bone surface was physically cleaned and they drilled to obtain powdered bone (usually around 500 mg, but see Table 5).

In order to obtain purified collagen, the bone powder was then subjected to acid-base-acid washes, 0.5M HCl, followed by 0.1M NaOH and then by 0.5M HCl, with multiple distilled water rinses done between each step. For more specific details see Brock et al. (2010). This was followed by gelatinization of the collagen in hot acidic solution (75°C for 20 h). The gelatin obtained was passed through a regular filter and an ultrafilter, the latter to remove low weight components (<30 kD) that are most likely to be contaminants and/or degraded collagen (Higham et al., 2006). It was then centrifuged at 2500-3000 rpm. After being freeze-dried, it was combusted, graphitized and measured (Dee and Bronk-Ramsey, 2000; Bronk Ramsey et al., 2004b; Brock et al., 2010).

When the samples were first analyzed (2008), ORAU was using a background correction that was subsequently improved in 2010 (Wood et al., 2010). Due to the old ages of the samples and small quantities of collagen extracted, dates were initially infinite. Once the new background correction was determined, however, dates were recalculated and became finite (see [Table 5](#)).

## 7. Results

Unfortunately, none of the Laouza or Salpêtrière samples had sufficient collagen for dating (see Table 4). For Esquicho-Grapaou, of 6 samples attempted, 2 produced dates (Table 5). For those that produced a date, yields (in mass and percentage) are on the low side, indicative of poor collagen preservation, also seen by the fact that other samples failed to produce enough yield. All other parameters are within good ranges, however. Results are presented in conventional, uncalibrated radiocarbon years BP as well as calibrated form. Calibration was done with OxCal 4.3.2 (Bronk Ramsey, 2009) based on IntCal13 (Reimer et al., 2013).

The two samples that had sufficient collagen produced statistically overlapping results. In addition, one of them (Esq 75 T22 #82) was dated twice as part of normal laboratory procedure (35 900 ± 800 (OxA-21716) and 36 500 ± 1000 (OxA-21732)). The ORAU lab routinely, randomly, selects some samples to date twice as a verification of their system. Its two results are statistically equivalent. As this double dating pertains to the same bone, we combined these two results with the Combine function in OxCal, obtaining 36 150 ± 626 BP (41 933-39 542 cal BP at 95.4%), as shown in Table 5. The other sample (Esq 78 T23 #9) gave 35 550 ± 750 (OxA-21717) (41 651-38 700 cal BP at 95.4%).

## 8. Discussion

In recent years a number of Protoaurignacian (PA) assemblages from western Mediterranean Europe were dated using a similar approach to that used at Esquicho-Grapaou (Szmids et al., 2010a; Barshay-Szmids et al., 2012, 2018b; Wood et al., 2013, 2014, 2018). These included dating taxon-identified organic samples and ensuring that dated samples bore direct evidence of human modification/involvement, sampling from zones identified as stratigraphically more reliable, examining chemical and elemental values of results to ensure that everything was within reliable and normal ranges (percentage and weight collagen, %C, C/N ratio,  $^{13}\text{C}$ ,  $^{15}\text{N}$ ) and using more stringent pretreatment cleaning methods (ABOX-SC, ultrafiltration) when feasible. These better-dated PA assemblages were summarized and plotted in a recent paper (Barshay-Szmids et al., 2018b: Tables 3a, 3b, Figures 7a, 7b).

In that study, it was shown that the modelled Protoaurignacian start date in southwestern Europe (based on Isturitz, Pyrenean France) was 42.8-41.3 ka cal BP, clearly before Heinrich Stadial 4 (HS4)/Glacial Stage-9. This is independently supported by a number of Protoaurignacian assemblages in Italy (Serino, Castelcivita) having been found under the Campanian Ignimbrite ash (dated to 39.99-39.71 ka at 95% confidence interval by  $^{40}\text{Ar}/^{39}\text{Ar}$  (Giacco et al., 2017)), a major volcanic eruption that occurred at the onset of HS4.

We replotted these recently-dated PA dates with the inclusion of the two new Esquicho-Grapaou dates. (see Barshay-Szmids et al., 2018b Tables 3a, 3b for all raw data, to which is added the data presented here from Esquicho-Grapaou). For the graph of dated PA sites across Western Europe using improved protocols, we count a total of 52 dates from 13 separate PA levels (1 layer each from 13 sites). These are plotted as calibrated (unmodelled) dates. From these, one can see that the PA ends by 35 ka cal BP (Fig. 9a), or much earlier (around 38 ka cal BP) if only looking at the more selective criteria for assemblage inclusion (i.e. removing assemblages bearing controversy: see Barshay-Szmids et al., 2018b for discussion) (Fig 9b). The Esquicho-Grapaou dates (in red) fall in the middle section of these recently-dated PA dates of Western Europe. The Esquicho-Grapaou dates are the first AMS  $^{14}\text{C}$  dates of the PA in Mediterranean France. All other dated PA sites are either in different parts of France (Pyrenean, Northeastern) or outside France (Barshay-Szmids et al., 2018b, Table 3a). The oldest dates come from the Isturitz site and the most recent are L'Arbreda H, Mochi G and Les Cottés 04 lower (Fig. 9a). In the more selective set (Fig. 9b) the latest ones are Fumane A2 and Serino 12.

Clearly the Protoaurignacian dates from southwestern Europe overlap fully with the last Neanderthal sites (dated to 41 030-39 260 modelled BP (95.4% confidence interval) (Higham et al., 2014). They also overlap partially with the range of Châtelperronian dates (44 000-40 000 cal BP) (Hublin et al., 2015) and Uluzzian dates (45 000-39 000) (Douka et al., 2014).

Until fairly recently, the Châtelperronian and Uluzzian were thought to be the work of acculturated Neanderthals who had modified their behaviour based on viewing the material culture of, and/or interacting with, *Homo sapiens*. More recently, however, two deciduous hominin molars from Uluzzian layers at Grotta del Cavallo were attributed to *Homo sapiens* (Benazzi et al., 2011), so this technocomplex has now been associated with them (although see Zilhão et al., 2015). At the two sites where a Neanderthal-Châtelperronian link has been proposed (Saint-Césaire and Grotte du Renne), recent lithic re-assessments, refitting, new dating, and detailed taphonomic work have called this association into question, arguing for admixture as the source of the association (Bar-Yosef and Bordes, 2010; Higham et al., 2010; Gravina et al., 2018). Yet others feel that there is no strong evidence against the idea that Neanderthals are associated with the Châtelperronian at these sites (Caron et al., 2011;

Hublin et al., 2012; Welker et al., 2016). The argument either way for the Uluzzian and the Châtelperronian maker(s) is based on very few sites, so we should stay open to various scenarios.

The Early Aurignacian in Central Europe (Higham et al., 2012; Nigst et al., 2014) also represents an early incursion of modern humans, with dates very similar to the earliest PA dates. Other Initial Upper Palaeolithic (IUP) industries in Central and Eastern Europe are likely made by AMHs and predate the Châtelperronian and Uluzzian (Hublin et al., 2015). In other words, the PA is unlikely to have been the first incursion of AMHs into Europe, but it is among the early ones.

In Mediterranean France, though, the Protoaurignacian is currently the first evidence we have of AMHs dispersal, as there are no Châtelperronian, Uluzzian or IUP assemblages. A techno-complex named Neronian has been proposed as a transitional industry in the Rhône Valley, found at a number of sites including Grotte de Néron and Grotte Mandrin (Combier, 1967, 1990; Slimak, 2007). The hominin responsible for this industry is unknown and radiocarbon dates could not be obtained (lack of collagen or beyond the radiocarbon range) (Higham et al., 2014: Supp. Info).

On a regional scale, early modern humans clearly shared a time slice with Neanderthals. Genetic evidence shows there was gene flow (Green et al., 2010) plausible with some overlapping dates. According to one large-scale study across Europe (Higham et al., 2014) the two populations could have co-existed for 2600-5400 years (this study included dates from only one Mousterian site in Mediterranean France, so one cannot conclude regionally). Now that we know the dates of the PA at one site in Mediterranean France (with another having failed), figuring out the extent of Neanderthal-AMHs overlap in this particular region would require dates on its few other PA sites (Onorardini, 1986, 2004; Bazile and Sicard, 1999; Bon, 2002; Slimak et al., 2006), as well as detailed comparisons (with stratigraphic integrity assessments) of Mousterian and Early Upper Palaeolithic records at stratified sites in this region (Szmídt, 2003, 2009).

Thus, nearly fifty years after its unique and specific cultural attributes were first recognized and described, Esquicho-Grapaou again becomes the first, this time in showing us the timing of this important dispersal in the Mediterranean part of the southern dispersal route.

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### **Figure and Table captions**

Figure 1: Map of sites mentioned in the text and some other key Aurignacian sites of Western Mediterranean Europe.

Figure 2: Site plan of Esquicho-Grapaou, showing layout of early 20<sup>th</sup> century excavations and Bazile rescue excavation (squares R-V and 11-22). T23 (not shown here) represents only a small portion (bench, close to the wall of the cave) along the T22 square.

Figure 3: Stratigraphy in squares T20-T22, Bazile excavation, Esquicho-Grapaou, showing the Mousterian (CC2), Protoaurignacian (SLC 1a+b) and Early Aurignacian (BR1) layers. Triangles, stars and numbers correspond to palynological samples.

Figure 4: Lithic industry from Esquicho-Grapaou level SLC1a (Protoaurignacian). 1. Appointed blade; 2, 4, 5, 11: simple endscrapers; 3: retouched blade; 6, 7, 8: truncated pieces; 9: naturally-backed knife; 10: burin on an oblique retouched truncation

Figure 5: Dufour bladelets from Esquicho-Grapaou level SLC1a (Protoaurignacian)

Figure 6: Photo of the ivory shaped bead from Esquicho-Grapaou, level SLC1a. (Photo by P. Jugie, *Musée National de Préhistoire*)

Figure 7: Diaphysis splinter from Esquicho-Grapaou, level SLC1b (T22#158, detail) attributed to *Equus* with green fracture and several cut-marks; dimensions (mm) Length=75.9; width=25.2; cortical thickness=10.5 (scale: white line/square = 1 cm)

Figure 8: Diaphysis splinter from Esquicho-Grapaou, level SLC1 (U23#27) attributed to *Equus* with many cut-marks; dimensions (mm) Length=45.9; width=16.5; cortical thickness=12 (scale: white line/square = 1 cm)

Figure 9a: Plot of recently-dated AMS <sup>14</sup>C-dated PA assemblages, in calibrated form using OxCal 4.3.2 (Bronk Ramsey, 2009). Esquicho-Grapaou dates are in red. Further information about these samples is provided in Barshay-Szmidt et al. 2018b, Table 3a.

Figure 9b: Plot of recently-dated AMS <sup>14</sup>C-dated PA assemblages pruned to exclude sites considered controversial by some (see text for details), in calibrated form using OxCal 4.3.2 (Bronk Ramsey, 2009). Esquicho-Grapaou dates are in red. Further information about these samples is provided in Barshay-Szmidt et al. 2018b, Table 3b.

Table 1: de Sonneville-Bordes and Perrot lithic type-list of Esquicho-Grapaou layer SLC1a and La Laouza layer 2b1. Translated names in English are from Sisk and Shea, n.d. (downloaded Feb. 10, 2020, from [www.researchgate.net/publication/273911029](http://www.researchgate.net/publication/273911029)).

Table 2: Faunal list of the SLC1 level from Esquicho-Grapaou. NR: number of remains; NISP: number of identified specimens; MNI: minimum number of individuals; Age: juv=juvenile; ad=adult; K=carnivore actions; H=hominid actions (includes cut-marks, chop-marks, notches and flaked bones); green μ= green breakage without carnivore marks; burnt=burnt bone, mostly black. In the sieving, 90.6% of faunal remains are < 2 cm in size.

Table 3: Previously done radiocarbon dating (conventional method) at Esquicho-Grapaou, based on samples from Bazile 1970s rescue excavation. QM: Quina Mousterian; PA=Protoaurignacian; EA=Early Aurignacian. Laboratory codes: MC=Monaco Radiocarbon Laboratory; Ly=Lyon Radiocarbon Laboratory

Table 4: Information of the bones submitted for radiocarbon dating from l'Esquicho-Grapaou, La Laouza and La Salpêtrière in this project, including provenience and taxonomic information about each. Samples whose collagen extraction was attempted but which did not produce a date are included. Archaeological cultures were assigned based on lithic analyses in the strata: PA=Protoaurignacian; EA=Early Aurignacian. Laboratory codes are OxA (ORAU) and TO (IsoTrace). As the project progressed, sample lengths became standard part of protocol. For samples chosen prior to that, lengths had to be estimated after the fact through examination of sample photographs (ruler was in photograph). The latter are thus labelled as approximate.

Table 5: Results of the successfully-dated Esquicho-Grapaou bones in this project, including radiocarbon, yield and stable isotope results. Calibration of dates was done with OxCal 4.3.2 (Bronk Ramsey, 2009) based on IntCal13 (Reimer et al., 2013). Stable isotope ratios are expressed in ‰ relative to the VPDB standard for carbon and the AIR standard for nitrogen.

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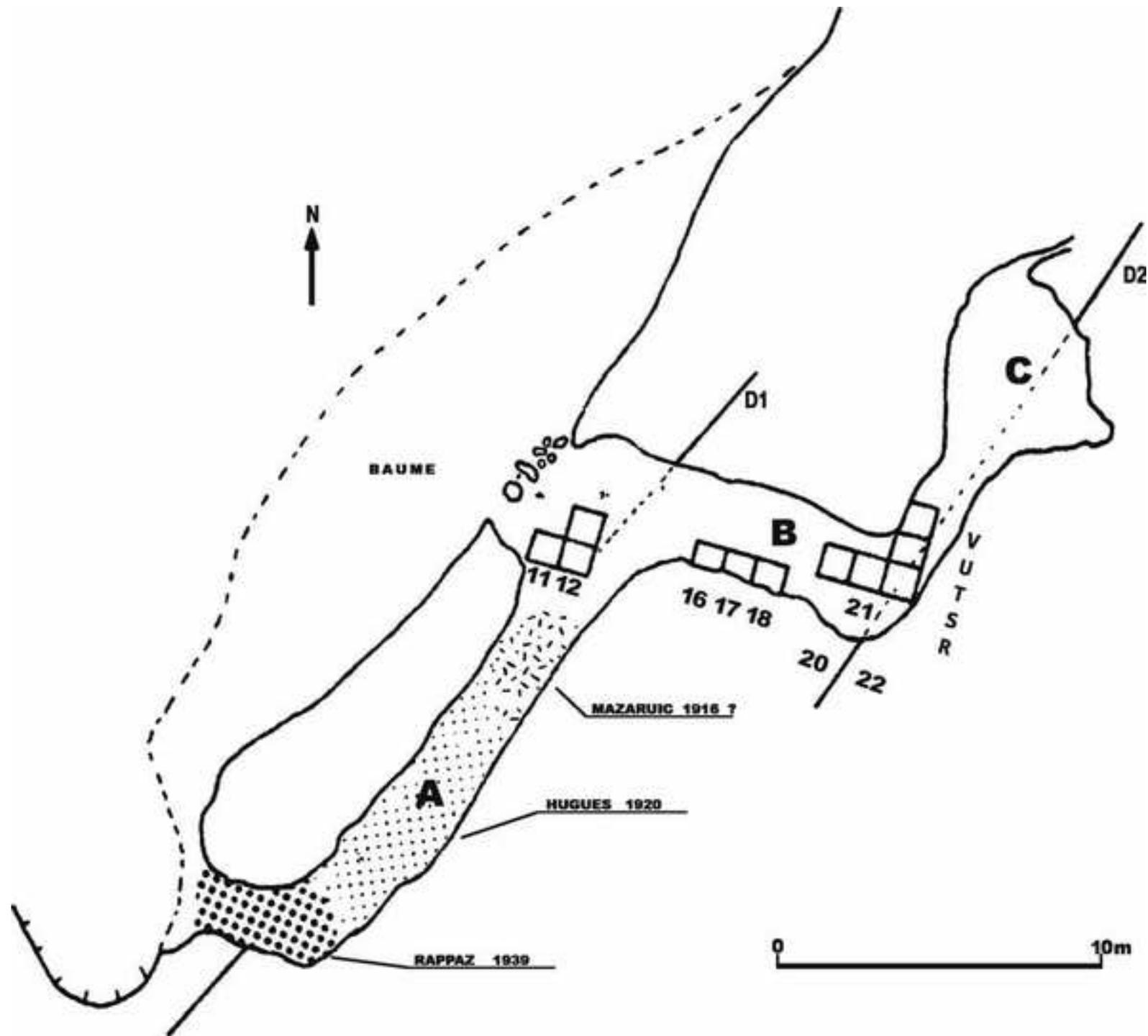
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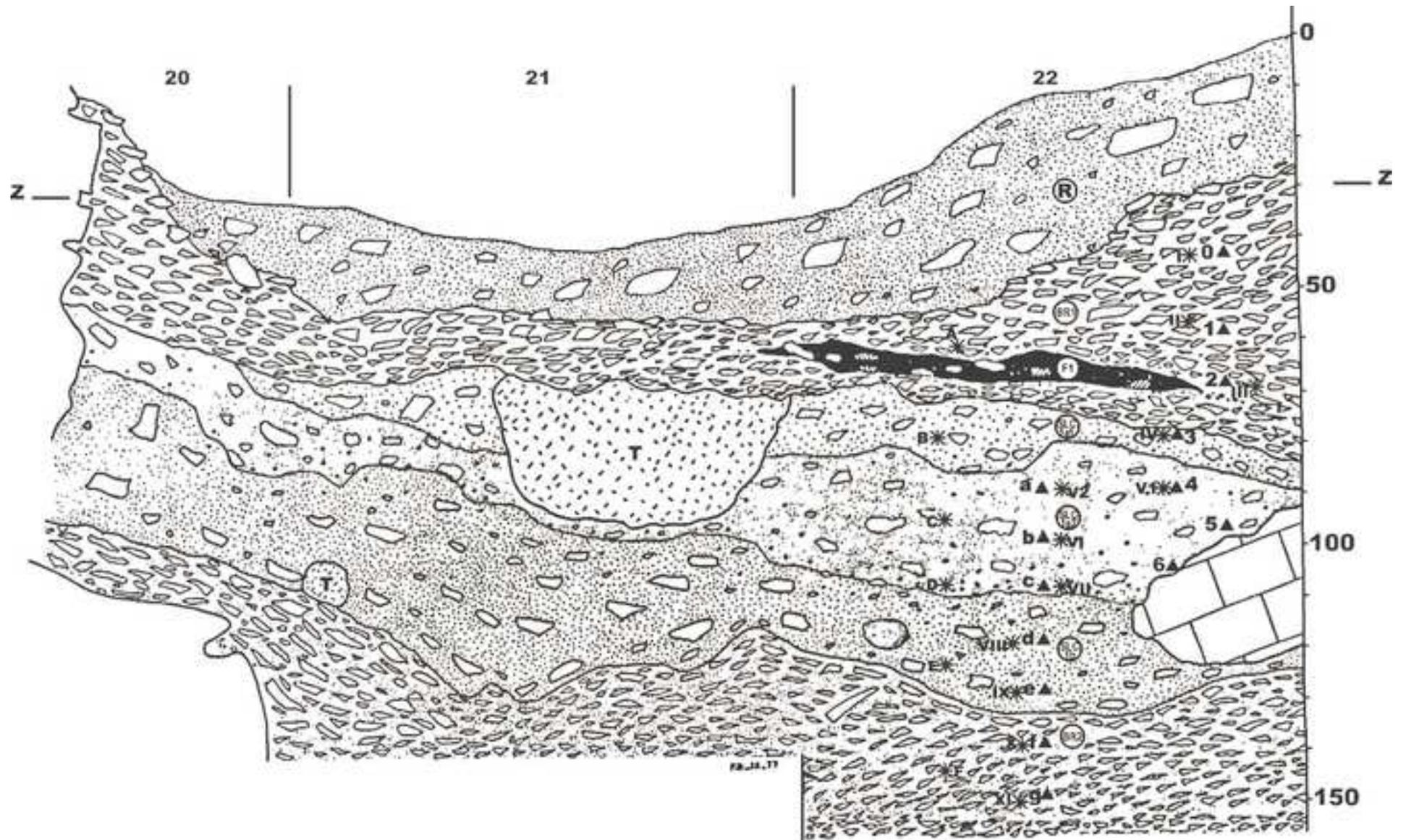
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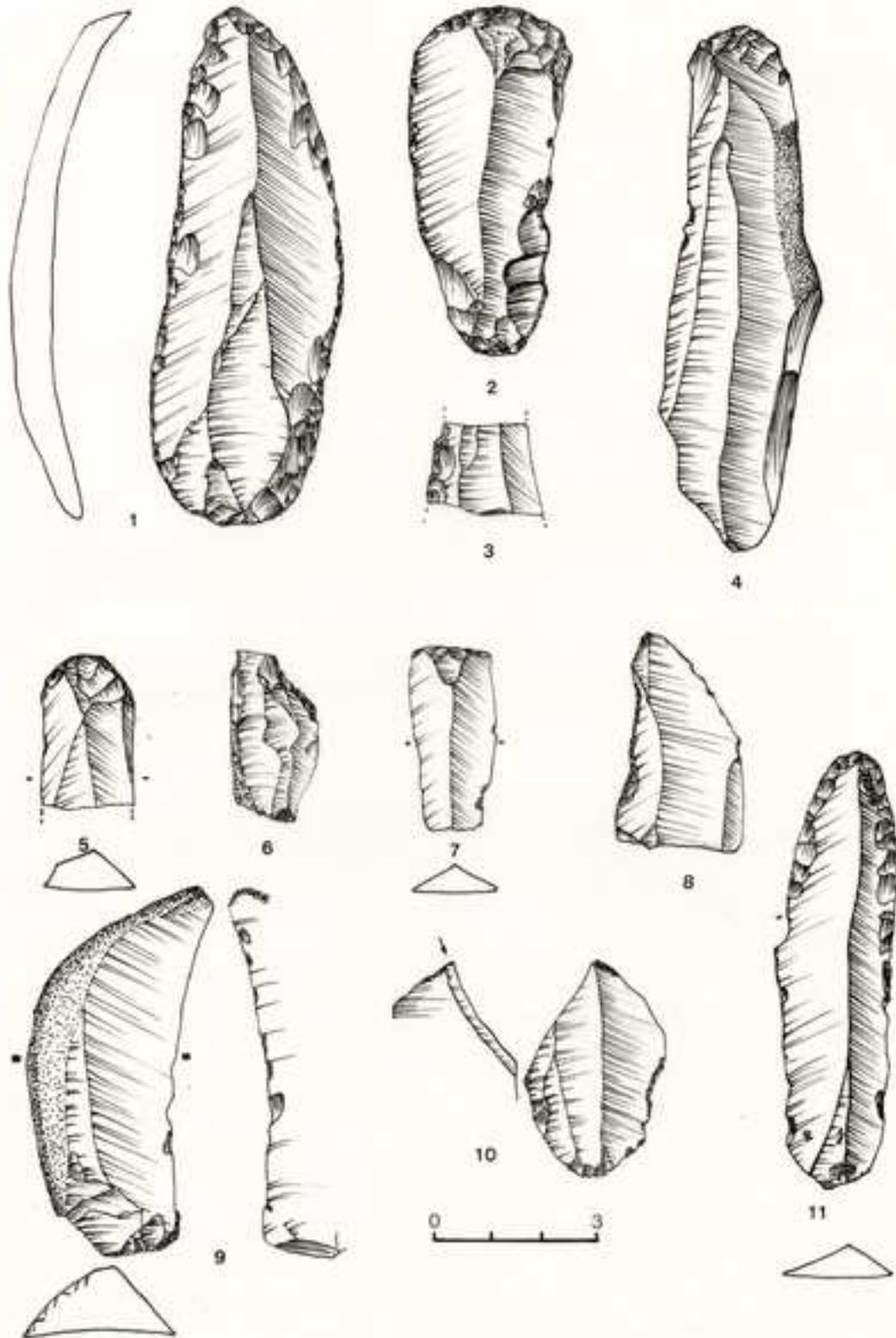
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Figure 1









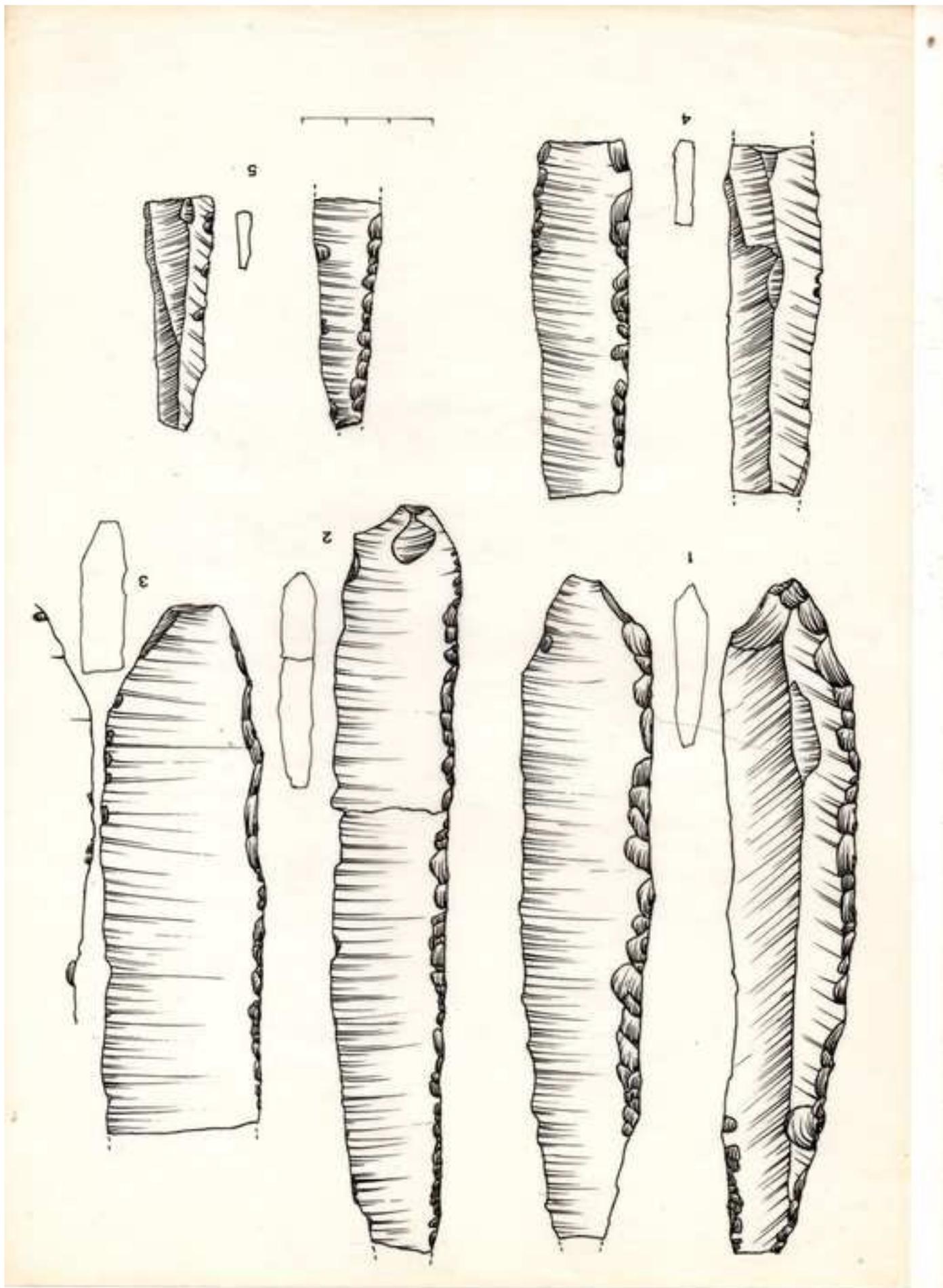


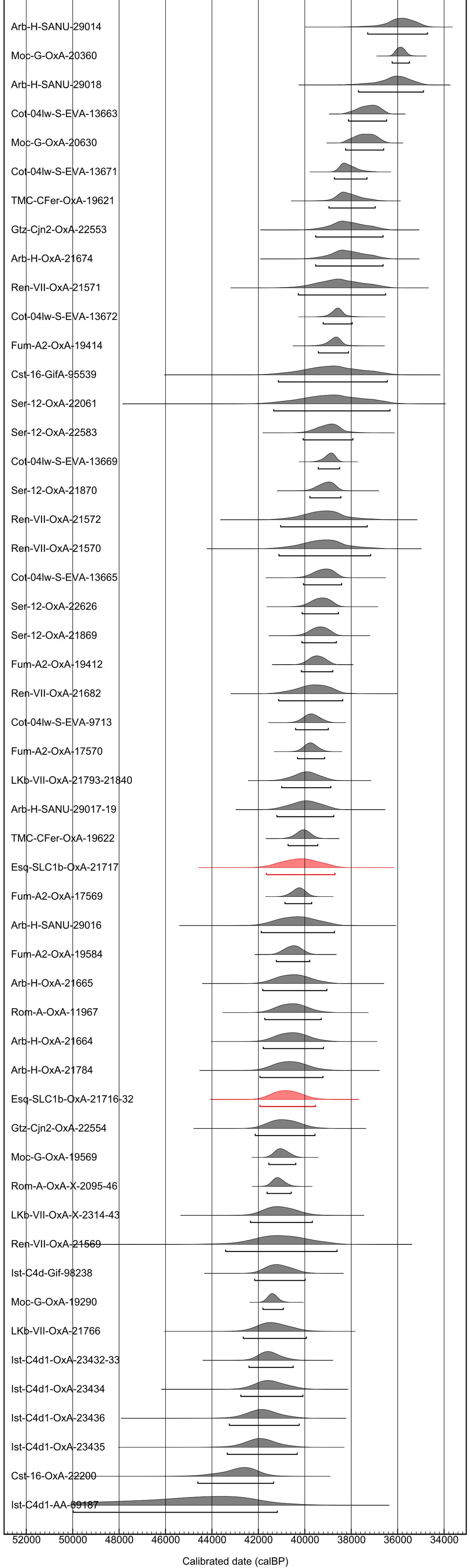




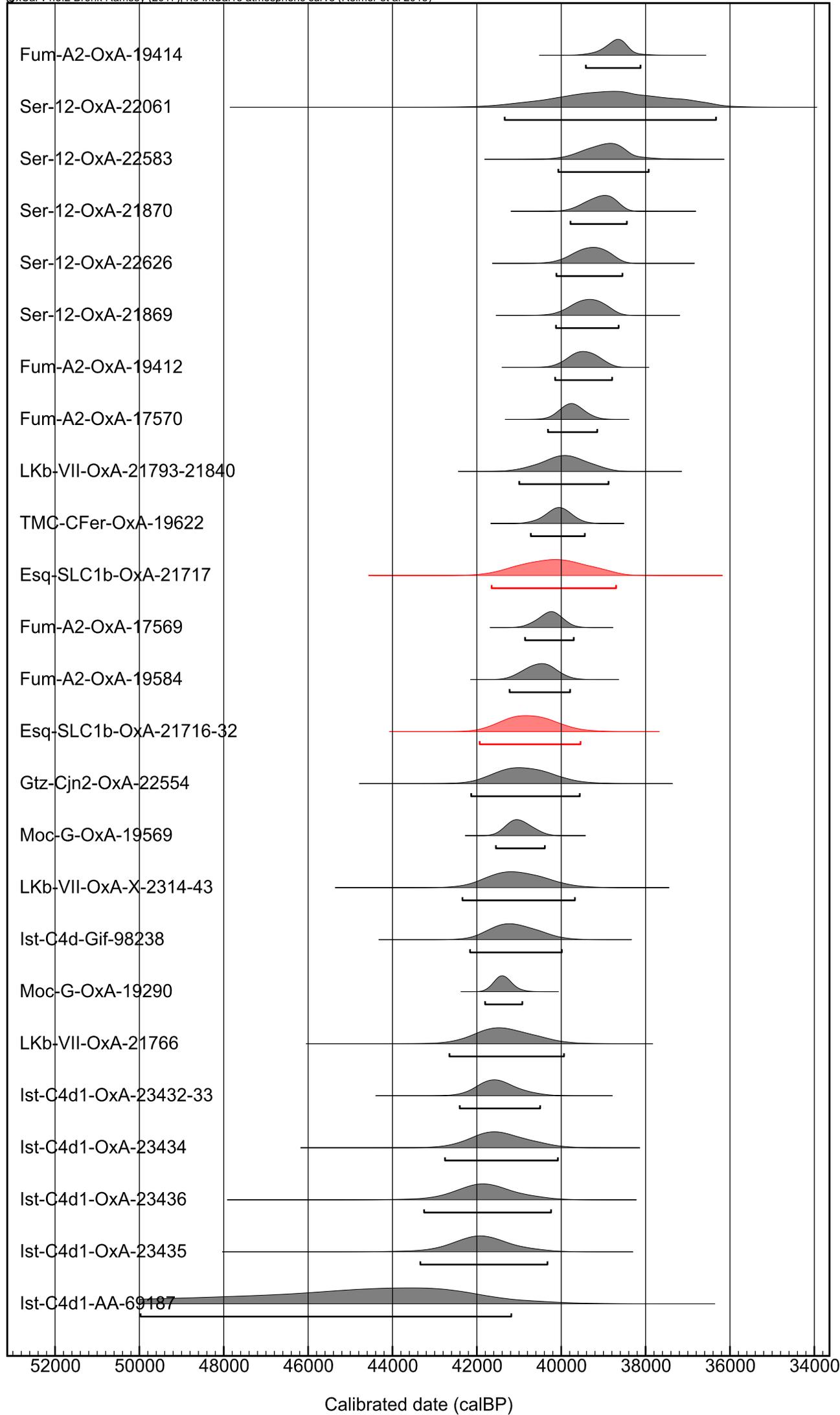
Figure 8

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Calibrated date (calBP)

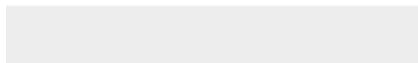
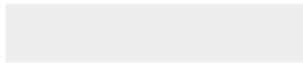




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**Table**

Tbl1-CBS-Esq-Typol-list.xlsx

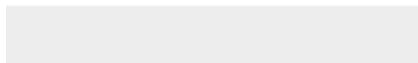




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**Table**

Tbl2-CBS-Esq-FaunaList.docx





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**Table**

Tbl3-CBS-Esq-Prev-dates.xlsx

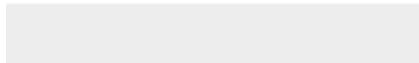




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**Table**

Tbl4-CBS-Esq-Sample-info.xls





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**Table**

Tbl5-CBS-Esq-DatingResults.xls

