DATING OF THE MIDDLE TO UPPER PALEOLITHIC TRANSITION AT THE ABRIGO 3 DEL HUMO (MÁLAGA, SPAIN)

Julián Ramos Fernández[i], Katerina Douka[ii], Alistair W. G. Pike[iii], Louise Thomas[iv], Peter van Calsteren[iv] y João Zilhão[v]

ABSTRACT: The Humo rockshelters contain one of the few long archeological stratigraphies spanning the Middle-to-Upper Paleolithic transition in southern Iberia. Stalagmite, bone and shell samples from Area 1 of the Abrigo 3 were collected in order to assess the chronology of the latest Mousterian of this *locus*. The results indicate that the sequence is older than hitherto thought, with U-series placing the level 25 stalagmite in MIS 5 and Radiocarbon on mussel shell placing level 18 in the range of 43.6-44.8 thousand calendar years ago. This latter result provides a *terminus post quem* for Level 17, the uppermost unit of the Abrigo 3 containing stone tools of Middle Paleolithic technology.

KEYWORDS: Iberia, Middle Paleolithic, Upper Paleolithic, Radiocarbon, U-series.

CRONOLOGÍA DE LA TRANSICIÓN DEL PALEOLÍTICO MEDIO AL SUPERIOR EN EL ABRIGO 3 DEL HUMO (MÁLAGA, ESPAÑA)

RESUMEN: Los abrigos del Humo contienen una de las pocas estratigrafías arqueológicas abarcando la transición del Paleolítico Medio al Superior en el sur de la Península Ibérica. Se han obtenido muestras de estalagmita, hueso y concha del Área 1 del Abrigo 3 para determinar la cronología del último Musteriense de este *locus*. Los resultados indican que la secuencia es más antigua que lo hasta ahora pensado, puesto que el método de la serie del uranio sitúa la estalagmita del nivel 25 en el MIS 5 y el radiocarbono sobre concha de mejillón sitúa el nivel 18 entre hace 43.6 y 44.8 miles de años (de calendario). Este ultimo resultado proporciona un *terminus post quem* para el nivel 17, la unidad más alta del Abrigo 3 que aún contiene una industria de tecnología Paleolítico Medio.

PALABRAS CLAVE: Península Iberica, Paleolítico Medio, Paleolítico Superior, radiocarbono, serie del uranio.

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1. INTRODUCTION

The Humo rockshelters are located in the La Araña archeological complex, at the eastern end of the Málaga city limits (Figure 1). This karst locality features a number of sites that have yielded evidence of human settlement between the Middle Pleistocene and later prehistoric times, including Paleolithic parietal art at Cueva Navarro. The area is also of significant geological and paleontological interest because of the preservation of paleocliffs, abrasion platforms, raised beaches and associated continental deposits that have provided key evidence for the study of Quaternary sea level change in the Mediterranean[1].

The line of rockshelters known as *Abrigos del Humo* is excavated along the lowermost paleocliff, where the high sea level stand of Marine Isotope Stage (MIS) 5 is marked at an elevation of 0.9 m amsl by a *Strombus bubonius* beach. Archeological work carried out in the area since the early 20th century explored a number of deposits filling cavities along the base of the paleocliff, where they have been preserved from the effects of erosion, quarrying and road construction that, seaward, entailed their loss. Such deposits thus correspond to the truncated fills of caves and rockshelters whose original topographic configurations are in most cases difficult if not impossible to reconstruct. Their archeological contents document use for settlement and ritual purposes in the Middle Paleolithic, the Solutrean, the Magdalenian, the Early Neolithic and the Copper Age[2].

Fieldwork in this locality was first carried out in 1965 by Gálvez Pacheco and has been ongoing since 1980 under the directon of one of us (J. R. F.). In 2003-04, the deposits preserved in

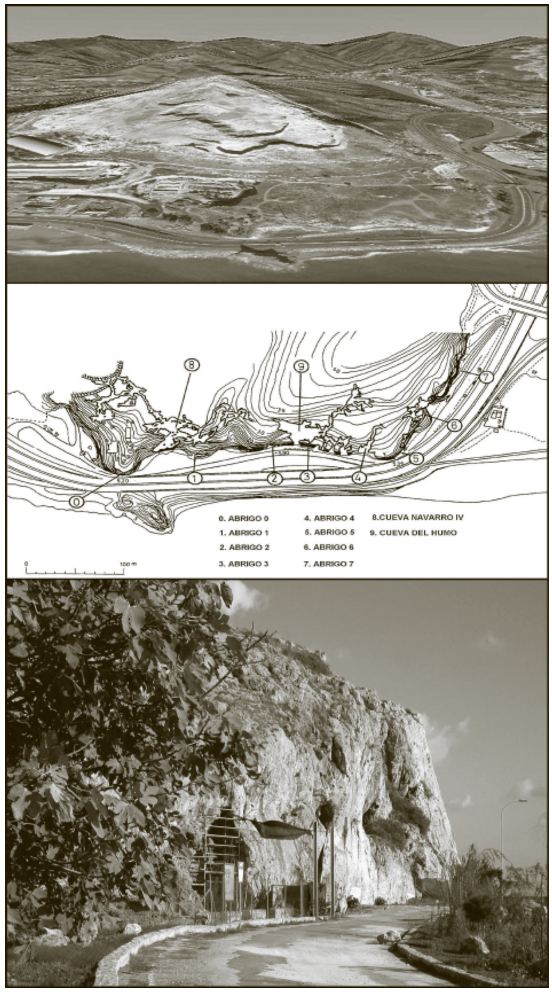

Fig. 1. Site location. Top: Google Earth view over the La Araña/El Humo archeological localities. Middle: Plan indicating the position of the different caves and rockshelters making up the complex. Bottom: View from the east over the paleocliff, the Cueva del Humo and the Abrigo 3 in December 2005.

the different areas of the Abrigo 3 were profiled with standard archeological methodology (Figure 2). The disturbed sediments were removed and systematically screened, the *in situ* deposits were excavated according to the observed stratigraphy and with plotting of individual finds, and samples for dating and for geological, palynological and anthracological analyses were

1 RAMOS-FERNÁNDEZ, J. y DURÁN, J. J. (1998); FERRE, E., *et al*. (2004).
2 RAMOS-FERNÁNDEZ, J. (2005); RAMOS-FERNÁNDEZ, J. *et al.* (2005).

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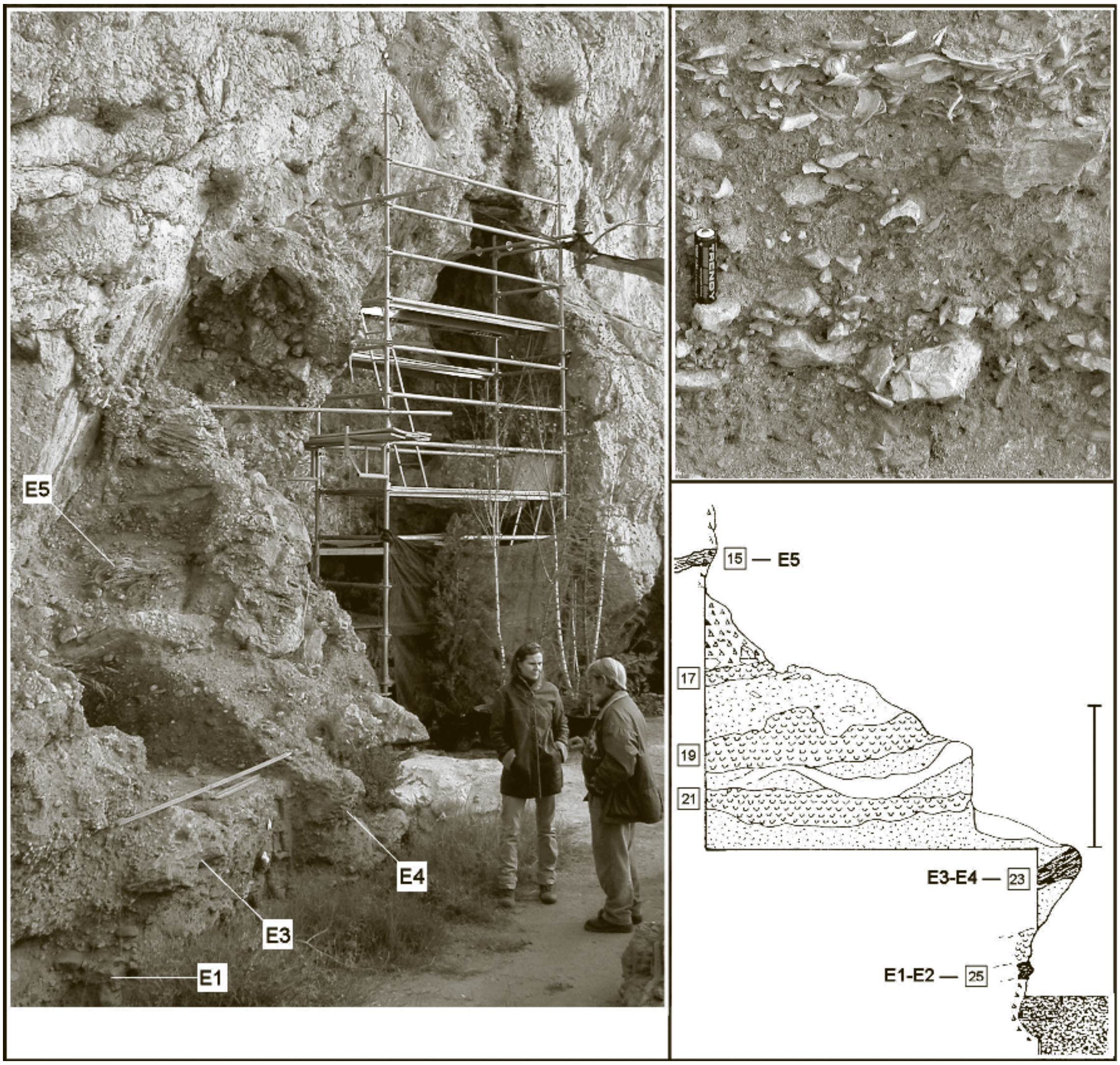

Fig. 2. The Abrigo 3. Left: General view from the east of the site in December 2005, with indication of the stalagmites sampled in the western area. Right, top: Detail of the stratigraphy in the east profile of the western area; note, at the top, the dense accumulation of osyter and mussel shells in level 19. Right, bottom: The stratigraphic succession observed in the east profile of the western area, with indication of the correspondence between stalagmite samples and stratigraphic units of the main shell-matrix deposits of MIS-3 age (levels 17, 19 and 21); the vertical bar marks the span of the deposits in the photo above.

taken, but, so far, only brief descriptions of the results obtained have been published[3]. The sedimentary fill consists of a succession of sandy deposits including wall-derived angular clasts and blocks and featuring interstratified stalagmite formations. The better preserved basal levels contain abundant stone tools associated with wood charcoal, animal bone and mollusc shells. In some levels, the latter form lenses of such density as to qualify for the category of shell-matrix deposits[4]; this is namely the case with Level 19 (Figure 2).

3 *Idem.*
4 *Sensu* Claassen: CLAASSEN, C. (1998).

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Of primary concern here is the observation that, in the western area of the site, the interface between the Middle and the Upper Paleolithic is located between Levels 15 and 17: Level 15 yielded Upper Paleolithic artifacts, the material in Level 16 is of uncertain affinities, upper Level 17 is a sterile eboulis deposit, and the stone tool component in lower Level 17 is unambiguously Middle Paleolithic. Unpublished Thermoluminescence (TL) results obtained on samples from stalagmites sandwiching these levels bracketed the transition to between ~23 ka (the age obtained for Level 15) and ~35 ka (the age obtained for Level 23).

As summarized in a recent review[5], the uppermost levels from the long Middle Paleolithic sequences excavated at Sima de las Palomas (Murcia), Gorham's Cave (Gibraltar) and Gruta da Oliveira (Portugal) are associated with dates younger than ~35 ka ^{14}C BP (~40 ka cal BP), while the Protoaurignacian of northern Spain and France, presumably modern human-associated, began as early as ~36.5 ka ^{14}C BP (~41.5 ka cal BP). The TL results reported for interstratified stalagmites from Humo 3 could therefore indicate that Levels 17-22 of the site represented yet another instance of the persistence of Neandertals and the Middle Paleolithic in southern and western Iberia.

However, the presence of an Evolved Aurignacian in Level 11 of Bajondillo (Torremolinos), a rockshelter site located some 10 km to the southwest, invited caution in the acceptance of such results. Although the radiocarbon dates for that level are problematic, as discussed in the review mentioned above[6], elsewhere in Europe the Evolved Aurignacian begins ~37 ka cal BP and is associated with modern humans exclusively[7]. Against this regional background, the *terminus post quem* for the overlying Middle Paleolithic represented by the unpublished TL age of ~35 ka for Level 23 of the Abrigo 3 implied that Middle Paleolithic Neandertals and Upper Paleolithic modern humans coexisted for several millennia in the Bay of Malaga; however, it could also be the case that the true antiquity of the Middle Paleolithic of the Humo complex fell significantly beyond the TL-derived estimates. It was in this context that it became important to assess the chronology of the Abrigo 3 del Humo with alternative techniques. Accordingly, the site was included in the 2006-07 dating project "The late survival of the Middle Palaeolithic and Neanderthals in the Iberian Peninsula" funded in the UK by NERC (Natural Environment Research Council) and AHRC (Arts and Humanities Research Council).

2. MATERIALS AND METHODS

In March 2006, one of us (J. R. F.) collected from the then extant profiles samples spanning the entire stratigraphic sequence of the Abrigo 3. In total, 9 speleothem samples and 71 bone and shell samples were taken from the three areas of the site. As the archeological stratigraphy was better defined in its westernmost part, Area 1, it was decided to focus our efforts on samples from this area: five speleothem samples were submitted for dating by U-series at the OUUSL (Open University Uranium Series Laboratory); nine bone and nine shell samples and were submitted for dating by radiocarbon at the ORAU (Oxford Radiocarbon Accelerator Unit, University of Oxford). The provenience of these samples is provided in Figure 3.

At the OUUSL, speleothem samples were spiked with a mixed ^{229}Th/^{236}U spike and totally

5 ZILHÃO, J. (2006).
6 *idem*.
7 ZILHÃO, J. (2007); HIGHAM, T. *et al.* (2011).

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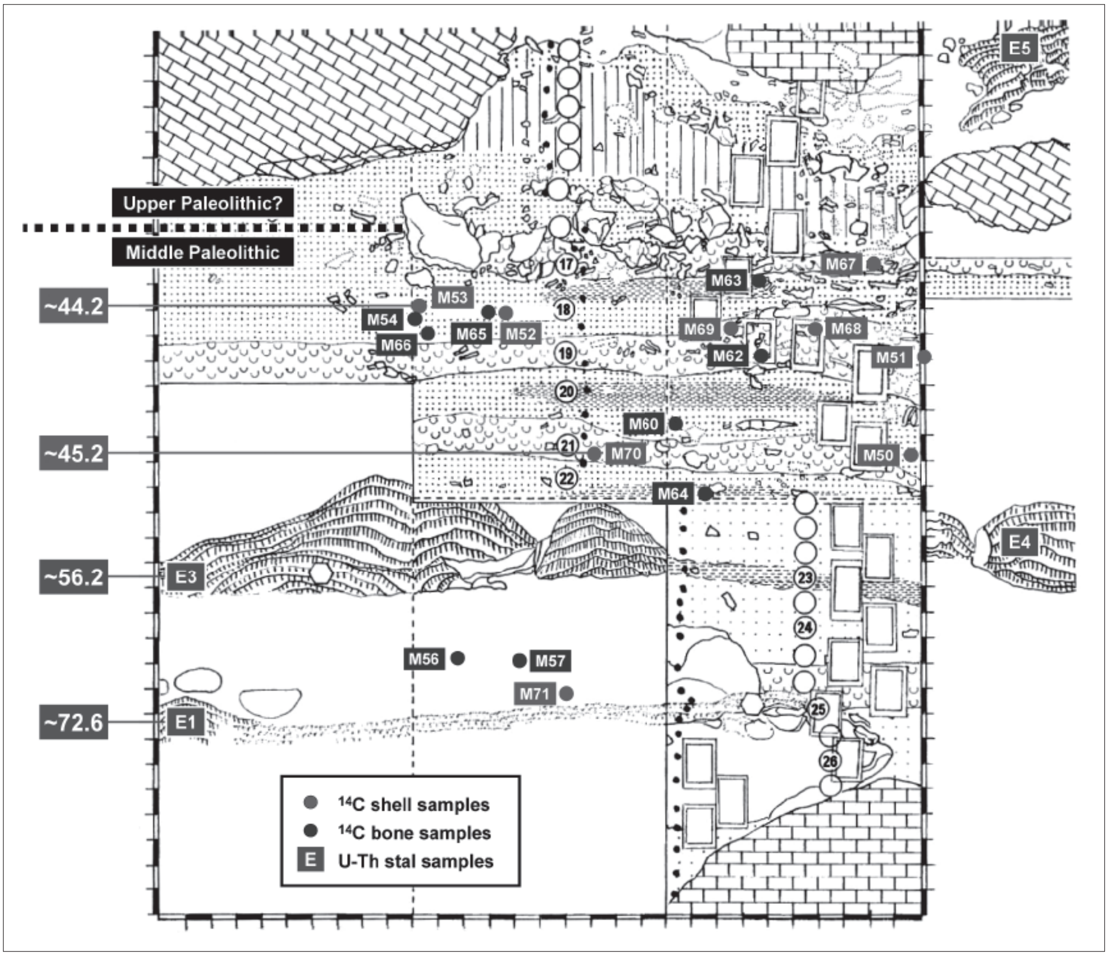

Fig. 3. The north profile of the western area of the Abrigo 3 with indication of the position of the samples collected from the profiles extant in March 2006 and of the approximate age obtained for the dated stratigraphic units. Sample E2, not shown, came from an area of level 25 located ~1.5 m to the east (i.e., to the right) of the profile illustrated here.

dissolved in a sequence of nitric acid and hydrofluoric acid. The uranium and thorium fractions were separated using standard ion exchange procedures (Calsteren & Thomas 2012). Uranium and thorium isotope ratios were determined on a Nu Instruments Multi-Collector Inductively-Coupled Plasma Mass Spectrometer equipped with an RPQ (Retarding Potential Quadrupole) lens for improved abundance sensitivity and Ion Counting. Fractionation and instrument drift were corrected using the data from the standard-sample bracketing protocol. Procedure blank contributions on ^{234}U and ^{230}Th were below ppm level. Ages marked 'corrected' were recalculated from (^{234}Th/^{234}U) and (^{234}U/^{238}U) ratios where the contribution of a 'detrital' component in secular equilibrium used a ^{232}Th/^{238}U crustal ratio of 3.12 and assumed that all determined ^{232}Th is detrital. Uncertainties for concentrations and activity ratios are 1 standard error, uncertainties in the calculated ages are 2 times the propagated uncertainties of the sample and bracketing standard runs and the uncertainty in the ^{232}Th concentration for the detrital corrected age. Decay constants are: λ ^{238}U=1.55125E-10; λ ^{232}Th=4.94750E-11; λ ^{234}U=2.82629E-06; λ ^{230Th}=9.15771E-06.

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Three shell samples were dated at the ORAU using routine pretreatment methodology for marine carbonates. The samples were surface-cleaned with an air abrasive system and using fine aluminium oxide powder as medium. Abrasion removed all adhering material and the first shell layers, until internal, usually opaque, shell layers were exposed. The cleaned shell underwent mineral staining using Feigl's solution. This way aragonite and calcite were visually separated and a small aragonitic fragment was sawed off and crushed with a mortar and pestle.

Approximately 50 mg of shell powder were reacted with 5 ml of 80% phosphoric acid (H_3PO_4) *in vacuo*. The CO_2 gas evolved via this process was purified in an EA-CF-IRMS system and was transferred to a graphitization rig where it was converted to graphite via H_2 reduction at 560° C for 6 h, in the presence of 2-2.5 mg of a pre-cleaned Fe^+ catalyst. The graphite was then pressed into an aluminium target holder, prior to measurement using an accelerator mass spectrometer (HVEE Tandetron AMS system).

The same procedure was followed for the reference carbonate sample IAEA C1 Carrara Marble, a geological sample of infinite age which is routinely analysed alongside the unknown-age samples to monitor introduction of modern contamination during sample pretreatment.

Despite the fact that other shell species were available for dating, we chose to date mussel shells and not limpets or scallops. Mussels are linked to food collection and are easy to harvest alive, therefore their age-at-death should reflect the time of collection. Moreover they are filter-feeders, which means that they feed on seawater dissolved inorganic carbon (DIC) and marine particulate organic matter (POM). Limpets were also collected for food since the Middle Palaeolithic, however they are sometimes suspected to incorporate into their shells dead carbon (containing no ^{14}C) from the limestone rocks on which they graze. Therefore, their measured radiocarbon content would require a particular correction, but the exact offset is unknown. Scallops, on the other hand, are deep-sea animals the collection of which for food purposes is difficult to prove. It is most likely that these shells represent beach collection and would therefore suffer from an inbuilt age. This means that the time of their collection (the archeological event of interest) and the time of their death (the event dated when measuring the shell's radiocarbon age) may be several centuries or even millennia apart.

Alterations in the mineralogy of an archaeological shell is usually reflected in the transformation of original aragonite and high-Mg calcite to secondary low-Mg calcite. Such diagenetic processes are very likely to affect the information we want to obtain especially from samples of such antiquity as the Abrigo del Humo shells. Limpets and scallops are almost entirely calcitic in nature, therefore presence of secondary low-Mg calcite would have been difficult to detect. Mussels contain both original aragonite and high-Mg calcite; in this study the aragonitic phase of the (nacre) was sampled and dated. This work was undertaken before a new pretreatment protocol was established at the ORAU for the physical separation of original aragonite from secondary, low-Mg calcite (CarDS)[8]. While we did not perform XRD for the precise identification of the dated mineral, staining with Feigl's solution allowed us to separate mechanically any potentially contaminating phase. Despite our efforts, the presence of contamination in the mussel nacre cannot be completely ruled out; we will return to this point in the next section.

8 DOUKA, K. *et al.* (2010).

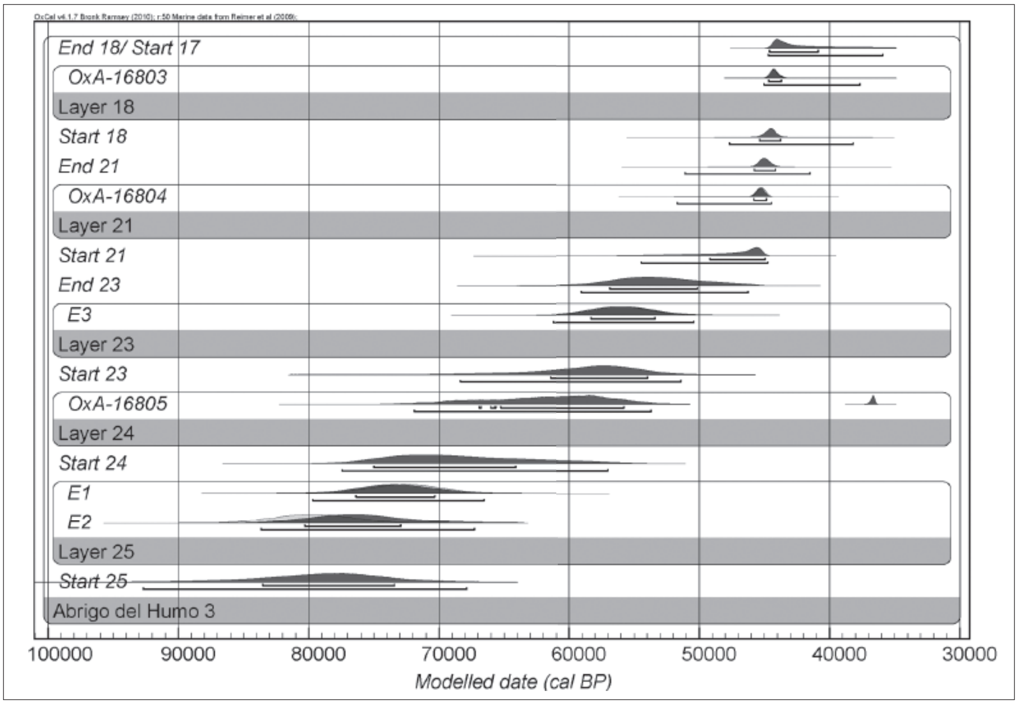

Fig. 4. Bayesian model containing the available chronometric data from Abrigo del Humo 3. The radiocarbon measurements were calibrated using Marine09 and a local reservoir offset. The U-Th determinations reflect calendar ages and do not require calibration. OxA-16805 was flagged an outlier and is marked in red (see text for details).

3. RESULTS

We obtained results for three speleothem and three shell samples, as reported and discussed here. The results and associated provenience and chemistry details are reported in Table 1 for the U-series speleothem dates and in Table 2 for the radiocarbon shell dates.

All radiocarbon results were calibrated using Marine09, which subtracts 400 ^{14}C years from IntCal09[9]. Using OxCal 4.1.7[10], all dating results, both U-Th and ^{14}C measurements, were included in a Bayesian statistical framework the results of which are presented in Figure 4. In this age model, the local reservoir offset of -22 ±35 ^{14}C years (CHRONOS database) was subtracted from each measurement prior to calibration. The calibrated results are reported at the 68.2% and 95.4% level of confidence (2 and 3σ). The small number of measurements do not allow any drastic refinement of the chrono-stratigraphic succession, however the model permits some statistically-sound observations to be made regarding the likely start and end boundaries of each dated layer, as well as on the duration of each phase (the archaeological levels, or Layers) and the identification of outliers.

Level	Sample #	Lab #	$\delta^{13}C$	Age BP	Age cal BP
18	M53	16803	-0.37	40730 ± 310	43590-44819
21	M70	16804	0.06	42140 ± 360	44580-45814
24	M71	16805	0.74	32580 ± 180	36325-37097

Table 1. OxA radiocarbon dates on Mytilus sp. shells from the Abrigo 3 del Humo. Calibration was carried out with the Calib 6.1.0 software using the marine09.14c curve.

9 REIMER, P. *et al.* (2009).
10 BRONK RAMSEY, C. (2009).

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Level	Sample Field #	Weight (mg)	^{238}U (ppb)	^{232}Th (ppb)	($^{230}Th/^{232}Th$) activity ratio	($^{230}Th/^{234}U$) activity ratio
25	E1	405.56	0.34942±0.00095	422.60±76.39	1.8707±0.0772	0.6797±0.0059
25	E2	525.31	0.22569±0.00032	55.21±9.98	7.6020±0.3147	0.5578±0.0049
23	E3	427.08	0.29711±0.00052	307.47±55.58	1.9108±0.0806	0.5950±0.0057

Table 2. OUUSL U-series dates on speleothem samples from the Abrigo 3 del Humo. Errors are 2 σ. See text for details on the correction for detritical contamination.

The U-series speleothem dates show that the deposition of the site's basal flowstone deposits (Levels 23 and 25) occurred significantly earlier than the unpublished 35 ka estimate previously available for Level 23 and the ~36.8-36.5 ka cal BP result (OxA-16805) obtained by radiocarbon for a mussel shell from Level 24. OxA-16805 is out of stratigraphic order and an outlier. It comes from a sample collected >1 m below Level 21, making it highly unlikely that the anomaly can be explained by post-depositional disturbance; nothing was observed in field that can corroborate such large scale movement of material. Dating marine carbonates close to the limit of the radiocarbon technique is not easy and requires careful sample selection, screening and cleaning. No XRD analyses were performed for the dated sample; therefore, diagenetic alterations and incorporation of younger carbon during recrystallization processes cannot be ruled out. Although the sample's chemistry provides no indication of contamination by younger carbonates (the $\delta^{13}C$ value does not reveal incorporation of lighter terrestrial carbon), in the context of the ensemble of results obtained we may conclude that OxA-16805 represents a minimum age only and, accordingly, should be interpreted simply as providing a *terminus ante quem* of 43.5 ka cal BP for the deposition of Level 24.

Even if a significant error is incorporated in the detrital correction and propagated to the corrected U-series age, stalagmite sample E2 (Level 25) remains firmly in the MIS 5 time bracket. Such an incorporation would imply a wider bracket for stalagmite sample E3 (Level 23), placing it in the region of 38 to 66 ka, but the radiocarbon results for overlying Level 21 constrain to 44.6 ka the upper limit of the age interval of E3. Overall, therefore, the results show that, in the stratigraphic sequence of Abrigo 3, only Level 23 and those above it are of MIS 3 age.

The ~44.8–43.6 ka cal BP radiocarbon date for Level 18 provides a *terminus post quem* for Level 17, the site's uppermost Middle Paleolithic level. However, as U-series dating of the speleothem sample from Level 15 was unsuccessful and no mussel shell samples were available from Level 17 itself, the age of this level cannot be firmly established at present.

Where the issue of the late persistence of the Middle Paleolithic is concerned, our results for the Abrigo 3 del Humo are, therefore, inconclusive. However, they corroborate the evidence concerning the Middle Paleolithic exploitation of marine resources provided by a cluster of other sites located along the Mediterranean coast of Iberia, between Gibraltar and Murcia[11]. The importance of such resources for the regional populations of Neandertals is attested by the fact that shell accumulations are important even in Level 24, which dates for bracketing speleothems place in MIS 4, i.e., in a glacial maximum, when the coast line was farthest from the site. That such accumulations are

11 ZILHÃO, J. *et al.* (2010); COLONESE, A. C. *et al.* (2011); CORTÉS-SÁNCHEZ, M. *et al.* (2011).

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Table 2 (Cont.).

Level	($^{234}U/^{238}U$) activity ratio	age (ka) uncorrected	age (ka) corrected
25	1.0705±0.0106	121981/+ 3180/-3214	72558/+2721/-2745
25	1.0561±0.0031	88263/+ 2253/- 2273	79737/+2875/-2908
23	1.0666±0.0053	97422/+ 2767/- 2795	56214/+2232/-2250

more important in the overlying levels of MIS 3 age (namely, Levels 21, 19 and 17) is therefore explained as a byproduct of sea level change and corresponding implications for site function and resource acquisition patterns. All other things being equal (namely where technology and settlement and subsistence systems are concerned), it is to be expected that the accumulation of marine resources at hunter-gatherer settlement sites will vary inversely to the distance separating them from the coast. The Abrigo 3 evidence fits such an expectation and, in context, strongly suggests that, by early Upper Pleistocene times, marine resources were being routinely exploited, possibly on a seasonal basis, and played a rather significant role in Neandertal economies of southwestern Iberia.

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