Supporting Information

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Description of Sites and Samples That Could Not Be Radiocarbon Dated

Bones from 11 sites were screened for the preservation of collagen. Nine sites yielded no datable bone and are described below. Site locations are given in Fig. S1 and the percentage of nitrogen (%N) contents are given in Fig. S2.

Cendres, 38°41′N 0°08′E (Teulada-Moraira, Alacant), Elevation 50 m. Excavations since 1995 have uncovered a rich sequence of Upper Paleolithic deposits (1). Beneath levels containing Gravettian assemblages, a potential initial Upper Paleolithic was uncovered in 2005. We aimed to date this and the overlying initial Gravettian occupation. The surfaces of bones were poorly preserved. Three bones without cut-marks or other evidence of butchery were sampled. All failed the %N screening protocol.

Mallaetes, 39°00′44′′N 0°18′04′′W (Barx, Valencia), Elevation 500 m. Mallaetes contains a small collection of Aurignacian remains, including a massive based point and a lozangic point that are indicative of the Evolved Aurignacian, at the base of an Upper Paleolithic sequence (2). The site was excavated by Pericot and Jordá between 1946 and 1949 and Fortea and Jordá cleaned sections and excavated a small area in 1970 (3). The majority of lithic and bone artifacts were found in the earlier excavations. The chronology of this assemblage is based on a single conventional radiocarbon date on a mixture charcoal and sediment (29690 ± 560 BP KN-1926, 36120–32920 calBP) from the 1970s excavation (3).

Given the age of Pericot and Jordá's excavation and the low occupation density, only modified remains were sought from the collection held at the Museu de Prehistòria de València. The two bone points, on display, were considered likely to have been conserved, and a third bone point fragment, not on display, was small (<1 g) and consolidated. One cutmarked bone was found, but failed the %N screening test. A second bone passed the %N test, but the single striation was not regarded as a convincing cut mark and was therefore not dated.

El Niño, 38°33′N 2°4′W (Ayna, Albacete), Elevation 800 m. Initially noted for its Upper Paleolithic rock art (4), El Niño was excavated in 1973 (5–7), uncovering a sequence containing a limited assemblage, likely to be Middle Paleolithic, below the lower eboulis (level 6). As part of a project aiming to re-excavate this poorly known Middle Paleolithic site lead by Alejandro Garcia (Universidad de Cantabria), fauna was sampled to provide a first estimate of the age of the deposit. Twenty-three bones were screened for nitrogen content, only two of which showed clear signs of anthropogenic modification. All failed the screening protocol.

Quebrada, 39°44′51′′N 0°59′35′′W (Chelva, Valencia). Excavations since 2006 at the rock shelter of Quebrada have uncovered layers containing Mousterian assemblages (8). Two radiocarbon dates on charcoal suggest that one combustion feature was created just before 42 ka calBP in the uppermost Mousterian level (layer III) (40500 ± 530 Beta-244003, 45250–43450 calBP: 43930 ± 750 BP Beta-244002, extends beyond limit of calibration curve). Nine bones were sampled from spits (capas) 5 and 2 to test the existing dates. The surface of the bones was too poorly preserved to identify cut-marks and all failed the %N test.

El Salt, 38°41′13′′N 0°30′32′′W (Alcoy, Alcant), Elevation 680 m. El Salt contains a stratified sequence of Middle Paleolithic layers with a rich material record including large lithic and faunal assemblages and numerous combustion structures constituting a succession of archaeological palimpsest deposits (11–14). In the basal layers (S.U.XI–XII) human occupation was intense, and starts to diminish in S.U.X, currently undergoing excavation. Between VIII and V lithics are scarce, but five Neanderthal teeth were found in layer V. A radiocarbon date on bone found close to these teeth was treated with the ninhydrin method (Table S6). The bone yielded little carbon, and so the date of 37100 ± 320 BP (code unknown, 42470–41430 calBP) may be an underestimate. Taken at face value however, the date suggests that level V was forming shortly before the arrival of anatomically modern humans (AMHs) in northern Iberia.

This study attempted to test the chronology of units VIII to V. Of 40 bones selected with Bertila Galván and Cristo Hernández (Universidad de la Laguna) at El Salt and the Museu Arqueològic Municipal d'Alcoi, eight had a marginal nitrogen content and five, including a Neanderthal tooth, passed the screening test. Radiocarbon pretreatment was attempted on three bones and one tooth, but none yielded enough collagen for dating. Polyvinal acetate consolidation of the remaining bones, applied at the time of excavation, was suspected after examination under a microscope and FTIR spectroscopy. Regrettably, therefore, no further work was possible on bone.

Sima de las Palomas, 37°47′59′′ N0°53′45′′W (Torre Pacheco, Murcia), Elevation 123 m. Sima de las Palomas is a karstic shaft that was mined in the 19th century. The Upper Cutting, excavated since 1992, contains at least four Neanderthal individuals associated with a Mousterian assemblage, which Walker et al. (9) claimed post-dates the first occurrence of the Aurignacian north of the Ebro. The radiocarbon dates were on burned bone and the U-series dates on bone that had reached equilibrium in uranium uptake, and both sets of dates must be regarded with caution. Given these problems, Walker et al. (10) have expressed concern that the chronology may be inaccurate. To test the age of this deposit a Neanderthal phalange was screened. Sadly, the phalange did not past the screening test.

Cueva Bajondillo, 36°37′10′′ N 40° 29′ 33′′ W, (Torremolinos, Málaga, Andalucia), Elevation 10 m. Cueva Bajondillo contains one of the few Evolved Aurignacian assemblages (layer 11) in southern Iberia, overlying a sequence of layers containing Middle Paleolithic industries (layers 17–14) (15). Layers 12 and 13 fill a depression in the top of layer 14, and both may contain redeposited material (Zilhão, 2006; see ref. 47). The site was subject to a rapid rescue excavation in 1989, but the standing section has since been extensively studied by Cortés Sánchez (15). Thermoluminescence dates on burnt flint are anonymously young throughout the stratigraphy, and the chronology is based on radiocarbon dates on charcoal/sediment removed from the standing section from levels 14, 13, and 11 (15). Considering their large errors, these dates fall broadly in line with the expected age of the Evolved Aurignacian and place the Middle Paleolithic beyond 42 ka calBP. However, the sample type is far from ideal and this chronology requires testing.

The few bone fragments recovered by Cortés Sánchez (15) from layers 14–11 of the standing section were examined. Fragments were small (maximum dimension < approximately 5 cm) and thin (< approximately 0.5 cm); they were not identifiable to species and were too degraded to preserve signs of anthropogenic modification. Given the extremely low chance of collagen preservation, the fragments were not screened for %N. The only alternative samples for radiocarbon are marine shells; however, none have been recovered from the standing section from the units of interest. Cortés Sánchez (15) noted that the excavated lithic assemblages from layers 14–11 appear to be mixed, and so these shells samples were not selected for dating.

Nerja, 36°45′44′′N 3°50′42′′W (Maro-Nerja, Málaga, Andalucia), Elevation 158 m. A series of six radiocarbon dates on charcoal from the Gravettian levels 13 and 12 suggest that Nerja contains one of the earliest Gravettian ages in southern Mediterranean Spain (16), with a Bayesian model predicting a start date of 31680–28654 cal BP at 95% significance. To test, and hopefully increase the precision of this boundary, 25 faunal bones from capas XIII, 13, and 12 were selected. Unfortunately, all of these bones contained less than 0.5%N.

Gorham's Cave, 36°07′N 5°21′W (Gibraltar), Elevation 14 m. Gorham's cave has been subject to three main excavations: by Waechter [1948–1954 (17, 18)], the Natural History Museum (NHM) [1995–1998 (19)], and the Gibraltar Museum [1997–ongoing (20)]. The first two excavations were toward the front of the cave and the most recent toward the rear. Radiocarbon dates have been obtained from all excavations. Those from Waechter's excavation were measured conventionally and removed from a section in 1957, long after the excavation had ceased and should be viewed with caution (21).

Eighteen samples of charcoal and two burned bones have been dated from the NHM excavation. All charcoal fragments were treated with the gentle RR protocol at the Oxford Radiocarbon Accelerator Unit (ORAU) (Table S6), with the exception of OxA-6075 from context 4d/22, which was pretreated with the ABA protocol. Evidence of occupation is exceptionally scarce within the layers containing the purported final Middle Paleolithic (contexts 11–24) (22). When first described, this sample was considered largely undiagnostic, poorly associated with the dated charcoal, and potentially mixed, containing a rejuvination flake from a prismatic core and Levallois flake (19, 22). Dates from this context, of around 30 ka BP can therefore only provide a *terminus ante quem* for the abundant Mousterian assemblages in level 18 and level 19, which both contain charcoal dated beyond 40 ka BP, as first suggested by Pettitt and Bailey (21). The most recent excavations have been dated with radiocarbon on 22 charcoal samples treated with an ABA protocol (20). These samples place the final Mousterian between 32–24 ka BP (*ca*. 36–29 ka calBP), although Finlayson et al. (20) suggest a minimum age of 28 or 24 ka BP (*ca.* 32 or *ca.* 29 ka calBP). Zilhão and Pettitt (23) doubt the reliability of the Finlayson et al. (20) dates, questioning both the ability of the pretreatment to remove contamination and the association of the charcoal fragments with the small assemblage of lithics.

We attempted to date modified bone from both the NHM and Gibraltar Museum excavations. Of 49 cut-marked bones screened, none contained sufficient nitrogen to attempt a date.

%N Screening Results

The %N contents of all bones screened in this study are presented in Table S1. These results are compared with %N contents of bones from northern Iberia, obtained using identical methods, in Fig S2. Preservation of collagen is significantly worse south of the proposed Ebro frontier, most likely because of higher temperatures.

Published Radiometric Dates from Jarama VI and Zafarraya

Both Jarama VI and Zafarraya have been dated before this study (Table S2). Bones from Zafarraya dated by Michel et al. (26, 27) were screened for nitrogen as part of this study (Table S3). The results closely match the carbon and nitrogen stable isotope and elemental data of collagen obtained by the ORAU at the time of dating. Collagen samples that did not meet the criteria set by Van Klinken et al. (47) also did not contain sufficient nitrogen to suggest success by dating using the ultrafiltration protocol. These dates should therefore be viewed with caution.

Critique of Dates from Middle Paleolithic/ Neanderthal Assemblages Thought to Be <42 k cal BP in Southern Iberia

See Table S4 for a critique of dates from Middle Paleolithic/Neanderthal assemblages thought to be <42 k cal BP in southern Iberia.

Critique of Dates from Radiometrically Dated Evolved Aurignacian Assemblages in Southern Iberia

See Table S5 for a critique of dates from radiometrically dated Evolved Aurignacian assemblages in southern Iberia.

SI Methods

All bone samples were screened for nitrogen content before radiocarbon dating. The majority of nitrogen in bone is contained within collagen, the fraction selected for radiocarbon dating, and so can provide in indication of whether a bone can be dated. When collagen constitutes less than 1 wt% of the bone, quality assurance indicators, such as the C:N ratio, %C, and $\delta^{13}C$ often suggest that the protein is of poor quality (either being degraded or contaminated) and not suitable for dating (47). Brock et al. (24, 48) have shown that when bone contains more than 0.8% nitrogen, more than 1% collagen could be extracted with the ultrafiltration protocol in around 70% of cases. The method is minimally destructive requiring 5 mg of bone powder obtained with a dentist's drill after removal of the bone surface. The sample is loaded into a tin capsule and the amount of nitrogen remaining measured in an automated carbon and nitrogen analyzer (e.g., Carlo Erba NA 2000) coupled to a continuous flow-isotope ratio mass spectrometer (CF-IRMS) (e.g., Sercon 20/20) (24, 48).

The use of an ultrafilter to remove the smallest contaminants from bone collagen was first suggested in 1988 (49). When applied to bone of Paleolithic-age, the process often produces ages that are significantly older than collagen extracted and purified with other procedures (50, 51). This increase in age suggests improved removal of contaminants because young contamination has a much larger effect on the measured age than ancient contaminants containing no ^{14}C (*ca.* 80 ^{14}C years for ancient vs. *ca.* 13000 ^{14}C years for 1% modern contamination added to a sample of 50000 BP).

Radiocarbon dating followed the methods described in Brock et al. (48). Bones were treated with the ultrafiltration protocol, denoted by the laboratory code AF. After sequential washing in HCl to remove the mineral phase, NaOH to remove base soluble organic components and HCl to remove secondary carbonates, the collagen was gelatinized. Large insoluble contaminants were then removed from the soluble gelatin with a 45- to 90-μm Eeze filter before ultrafiltration with a Vivaspin 15 30-kDa MWCO ultrafilter to remove the smallest contaminants. The residue was then freeze-dried. As a precautionary measure, if it was possible that glues or consolidants had been applied to the bone, a sequence of methanol, acetone, and chloroform washes were applied before pretreatment.

Collagen was combusted in an automated carbon and nitrogen analyzer coupled to a CF-IRMS as described for %N measurement, allowing measurement of carbon and nitrogen abundance and stable isotopes. The remaining gas was collected cryogenically for conversion to graphite using an iron catalyst (52) and radiocarbon dated by accelerator mass spectrometry (53).

Dates have been calculated according to the conventions of Stuiver and Polach (53, 54) and corrected for pretreatment and combustion backgrounds (55). Samples are defined as infinite when the $F^{14}C$ is less than twice its error from 0 and thus indistinguishable from background (54). The infinite date is equivalent to twice the size of the error. For example, if a sample has an $F^{14}C$ of 0.0022 ± 0.0012 (equivalent to $49200 \pm 4{,}400$ BP), it is regarded as infinite and ascribed a date of 0.0024 $F^{14}C$ or >48500 BP. In terms of the conventional ages that are presented here, any dates with an error of 4,000 ^{14}C years or greater are considered infinite.

A summary of methods and laboratory codes is given in Table S6. This table also contains descriptions of methods no longer in use at the ORAU, and methods mentioned in the text that have been used by other laboratories.

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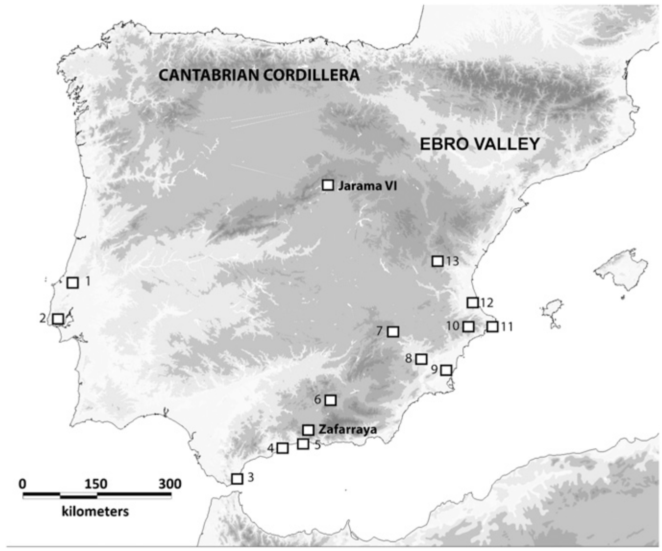

Fig. S1. The location of sites discussed in the main text and sampled in this study, alongside major topological features relating to the Ebro frontier hypothesis. 1, Gato Preto; 2, Pego do Diabo; 3, Gorham's Cave; 4, Bajondillo; 5, Nerja; 6, Carihuela; 7, El Niño; 8, Cueva Antón; 9, Sima de las Palomas; 10, El Salt; 11, Cendres; 12, Mallaetes; 13, Quebrada.

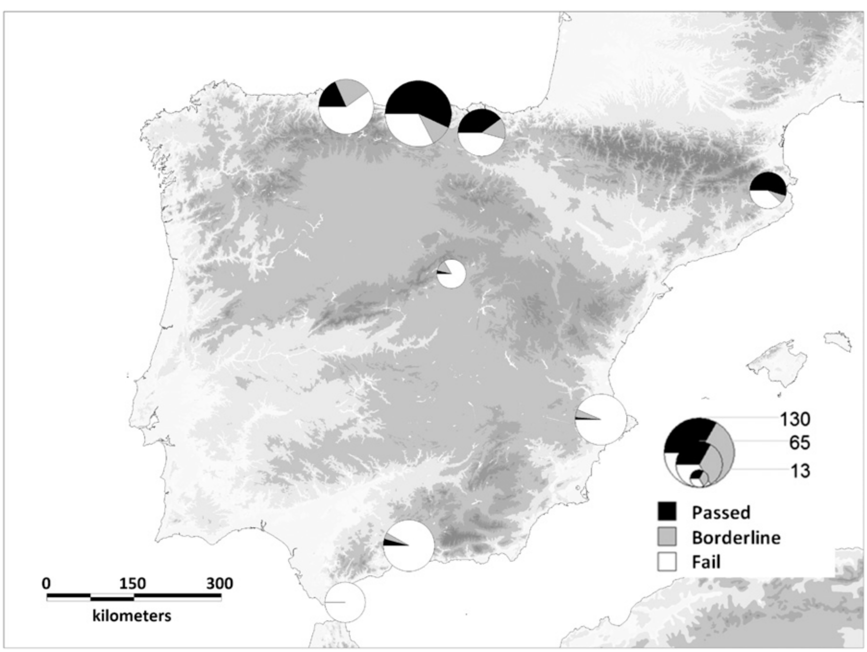

Fig. S2. The %N content of bone across Iberia. Nitrogen contents are higher in north of the Ebro, indicating markedly better preservation of collagen than in the south. Bones are ranked <0.5% N "fail," 0.5–0.7 "borderline," and >0.8% "pass" on the basis of how likely >1% collagen can be extracted from the bone with the ultrafiltration protocol (24). Each pie chart represents one administrative region. Data for southern Iberia is from *%N Screening Results* and northern Iberia from Wood (25).

Table S1. Results of the %N screening test for all samples in sites discussed in the main text and *Description of Sites and Samples That Could Not Be Radiocarbon Dated*

Table S1

If a sample of bone powder contains >0.7%N there is an 80% chance of the sample containing sufficient collagen for radiocarbon date (24) using the methods described in *SI Methods*. Samples containing 0.5–0.7%N are considered borderline here, and those containing <0.5%N were failed. Error on measurement is 0.2%N at 2 SD.

Table S2. Published radiometric dates from Jarama VI and Zafarraya

Table S2

14C denotes a conventionally measured radiocarbon date and AMS, a radiocarbon date measured by accelerator mass spectrometry. For laboratory pretreatment codes, please refer to Table S6. EU refers to an Electron Spin Resonance or U-Series date that assumes early uptake of uranium and LU, linear uptake. U-Series Alpha refers to U-Series dates measured by α-spectroscopy and TIMS, thermal ionization mass spectroscopy.

Table S3. The %N content of bones dated by Michel et al. (28, 29) and the quality assurance data of the dated collagen

Table S3

All but one sample have isotopic or elemental data that suggest collagen was very degraded or contaminated, and low nitrogen contents. To obtain a reliable radiocarbon date, bone should contain >1% collagen, $\delta^{13}C$ between −22‰ and −18‰, $\delta^{15}N$ between 2‰ and 12 ‰, C:N 2.9–3.4 and % C >30% (55). Error on stable isotope values is typically ± 0.2‰ and %N measurement ± 0.2%, 2 standard deviations.

Table S4. Critique of dates from Middle Paleolithic/Neanderthal assemblages thought to be <42 ka calBP in southern Iberia

Table S4

All uncalibrated dates are given at 1 SD, and all calibrated date ranges at 95% probability. Errors for nonradiocarbon dates are given at 1 SD.

Table S5. Critique of dates from radiometrically dated assemblages that are thought to be Aurignacian in southern Iberia

Table S5

All uncalibrated dates are given at 1 SD, and all calibrated date ranges at 95% probability. Errors for nonradiocarbon dates are given at 1 SD.

Table S6. Summary of radiocarbon pretreatment methods mentioned in the main text and *SI Text*

Table S6

Where a method is used at the ORAU (48, 56), the associated laboratory code is given.
*Method no longer in use at the ORAU.