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ESR/U-series chronology of the Neanderthal occupation layers at Galería de las Estatuas (Sierra de Atapuerca, Spain)

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Abstract:

The Galería de las Estatuas is a Mousterian site located within the Cueva Mayor-Cueva del Silo

karstic system at the Sierra de Atapuerca (Burgos, Spain). This site is characterised by an important Upper Pleistocene stratigraphic sequence in which a large number of lithic artefacts exhibiting clear Mousterian affinities and a rich assemblage of faunal remains have been found. Additionally, this site has yielded a foot phalanx of a clear Neanderthal affinities and its sediment has yielded Neanderthal mitochondrial and nuclear DNA.
Two test pits, GE-I and GE-II, have been excavated since 2008 which have been divided in 5 and 2 lithostratigraphical units, respectively. A minimum age of 45 ka cal BP for this site was obtained by radiocarbon dating in GE-II. This chronology was refined using single-grain Optically Stimulated Luminescence (OSL: 80 – 112 ka in GE-I and 70 – 79 ka in GE-II) and single-grain thermally transferred OSL (TT-OSL). Although in all case TT-OSL ages were systematically older than their single-grain counterparts. The detrital sequence is sealed by a flowstone whose base is dated to 53.7 ± 3.5 ka cal BP using U-series dating.
In order to better constrain the age of this site, nine herbivorous teeth were collected from the whole sedimentary sequence to be dated by the ESR/U-series dating method though only seven provided modelled ages. Six of them were collected from levels 2 to 5 of the GE-I test pit while the last tooth was taken from level 2 of the GE-II test pit. Our results, ranging between 80-110 ka, are in agreement with those obtained by single grain TT-OSL and suggest that the sedimentological levels containing Mousterian lithic artefacts and faunal and human remains began to be deposited during the second part of the MIS5. These results fill a temporal gap in the chronology of the Atapuerca sites for which no contemporary MIS5 date was obtained until recently.

Keywords: ESR/U-series dating; Neanderthal, Late Pleistocene; Atapuerca site complex; Iberian Peninsula

1. Introduction

The karst system of the Sierra de Atapuerca is well known for its Lower *(Gran Dolina* and *Sima del Elefante)* to Middle *(Gran Dolina, Galería* and *Sima de los Huesos)* Pleistocene and Holocene *(Cueva del Mirador, Galería del Sílex* and *Portalón)* paleoanthropological record (Apellániz and Domingo Mena, 1987; Carbonell et al., 2014). However, for a long time, no Upper Pleistocene chronologies were found in the caves, and the presence of

Neanderthal groups in the Sierra de Atapuerca was restricted to several open-air archaeological sites (Navazo and Carbonell, 2014) such as Hotel California (Arnold et al., 2013), Hundidero (Navazo et al., 2011), Valle de las Orquídeas (Mosquera et al., 2007) or Fuente Mudarra (Santamaría et al., 2021), among others.

The Galería de las Estatuas (GE) site, located in the upper level of the multilevel Torcas system, within the Cueva Mayor – Cueva del Silo complex (Ortega, 2009), revealed an Upper Pleistocene detrital sequence sealed by a speleothem (Aranburu et al., 2012; Arsuaga et al., 2017) in which the first Neanderthal fossil (a foot phalanx) in the Atapuerca karst system was found (Pablos et al., 2019), along with stone artifacts of clear Mousterian affinity as well as other palaeontological remains (Arsuaga et al., 2017). Furthermore, this site has also provided Neanderthal mitochondrial and nuclear DNA extracted from sediments which shows a population replacement in northern Iberia c. 100 ka BP (Vernot et al., 2021). Therefore, this site contains insights about Neanderthal occupations, subsistence strategies and population dynamics in the more inland Iberian Peninsula and completes the gap of Upper Pleistocene chronologies at the Sierra de Atapuerca karst system (Arsuaga et al., 2017; Vernot et al., 2021).

Most of the Neanderthal sites in the area surrounding the Sierra de Atapuerca are chronologically within MIS 3 – 4 periods with the exception of Fuente Mudarra site recently dated by OSL to MIS 3 – 5 (Santamaría et al., 2021). Inside the karst system, Galería de las Estatuas is the first archaeological site to be dated to MIS 4 – 5 using single-grain TT-OSL and OSL dating (Demuro et al., 2019). Taking into account the importance of this site within the entire Atapuerca karst system for understanding the time when the Neanderthal groups occupied the caves, a new chronological study was designed to further constrain the age of the Galería de las Estatuas site. The main aim of this study is to get the first ESR/U-series ages by dating several fossil teeth and provide a solid multiproxy and multi method dating approach for the stratigraphical sequence of both, GE-I and GE-II, test pits.

2. The Galería de las Estatuas (GE) site

The GE site is located in the upper level of the Torcas multilevel system (Cueva Mayor-Cueva del Silo complex), a sub-horizontal passage of about 615 m long, 10 m in width and 15 m in height, situated at +88 m above the current Arlanzón river (Ortega et al., 2013) (Fig. 1). It has been excavated since 2008 to present through two test pits called GE-I (ca. 9 m^2) and GE-II (ca. 6 m^2), revealing an Upper Pleistocene detrital stratigraphic sequence of, at

least, 5 and 2 lithostratigraphic units (LUs) in GE-I and GE-II test pits, respectively (Aranburu et al., 2012) (Fig. 2). It has been interpreted as an ancient entrance to the cave system that has been sealed by a stalagmitic flowstone (Arsuaga et al., 2017).

The five LUs have yielded a large number of lithic artefacts exhibiting clear Mousterian affinities and a rich assemblage of faunal remains dominated by equids and cervids (Arsuaga et al., 2017). Additionally, a human foot phalanx of clear Neanderthal affinities was found in the interface between LU-3 and LU-4 in the GE-I test pit (Pablos et al., 2019) and both pits have yielded evidence of Neanderthal ancient DNA (both nuclear and mitochondrial). Finally, the pollen analysis of the stratigraphical sequence also provided information regarding the paleoecological changes occurred in the Sierra de Atapuerca during MIS 4 – 5 (Arsuaga et al., 2017; Demuro et al., 2019)

Fig. 1. Approx. here

3. Previous chronological framework

The first attempt to date the GE site was carried out by applying radiocarbon dating (^{14}C) on faunal remains from levels LU-1 and LU-3. A series of eight ages were obtained ranging from 43.5 to > 46.3 ^{14}C ka BP (uncalibrated) but all these ages were close to the analytical limits of the technique and were cautiously interpreted as minimum ages (Arsuaga et al., 2017). A first U-series age of 13.7 ± 0.4 ka cal BP was obtained for the stalagmitic flowstone sealing the detrital sequence in GE-I and a second date from the base of the speleothem at GE-II yielded an age of 53.7 ± 3.5 ka BP (Martínez-Pillado et al., 2014; Vernot et al., 2021). Single-grain Optically Stimulated Luminescence (OSL) dating was also applied with the aim of better constrain the age of GE and seven samples were collected for this purpose: five from GE-I and two from GE-II. These single-grain OSL ages suggested that the sediment sequence excavated in GE-I test pit accumulated, at least, between 80-112 ka in GE-I and between 70-79 ka in GE-II. Replicate Thermally Transferred OSL (TT-OSL) single-grain ages were calculated and yielded results in agreement with the OSL chronologies at 2σ for three of the four samples investigated, although in all cases the TT-OSL ages were systematically older than their single-grain counterparts (GE-I: 123 ± 10 ka in LU-2 and 126 ± 14 ka in LU-4; GE-II: 86 ± 8 ka in LU-1 and 84 ± 7 ka in LU-2). These ages place the sedimentary sequence of GE-I within MIS 5 and the GE-II pit within late MIS 5 and early MIS 4 (Demuro et al., 2019).

Fig. 2. Approx. here

4. ESR/U-series dating of teeth

4.1. Sampling

Nine *Equus* molariform teeth were selected, coming from the 2008, 2009, 2010, 2012, 2014 and 2017 excavation campaigns (Fig. SM-1 in Supplementary Material). Seven of them (GE1801 to GE1807; see field labels in Table SM-2 in Supplementary Material) were collected from levels LU-2 to LU-5 of the GE-I test pit while two teeth (GE1808 and GE1809) were found at level LU-2 of the GE-II test pit (Table SM-2 in Supplementary Material).

4.2. Sample preparation

Teeth were prepared at the *Muséum national d'Histoire naturelle (MNHN)* of Paris (France) (Fig. SM-1 in Supplementary Material). Dental tissues (enamel, dentine and cementum) were mechanically separated and cleaned with an electric drill. In order to eliminate the effect of external α particles, a minimum thickness of 30-50 µm was removed from both, inner and outer, sides of the enamel fragments (Table SM-1 in Supplementary Material). Then, a part of the enamel was ground and sieved and two different granulometric fractions were collected. The 100-200 µm fraction was used for ESR measurements. The rest of the powder was employed for U-series analyses.

4.3. ESR analyses

The 100-200 µm grain-size clean enamel fraction of each tooth was split into 10 aliquots and 9 of them were irradiated with increasing doses (5, 10, 20, 40, 80, 160, 320, 640 and 1300 Gy) using a ^{137}Cs Gammacell gamma source at the *Centro Nacional de Investigación sobre la Evolución Humana (CENIEH)* in Burgos (Spain). ESR measurements were performed at room temperature at the *Institut de Radioprotection et de Sûreté Nucléaire (IRSN)* in Fontenay-aux-Roses (France) with an Elexsys E500 X-band spectrometer using the following parameters: 1 mW microwave power, 1024 points resolution, 100 kHz modulation frequency, 0.1 mT modulation amplitude, 20.48 ms conversion time, 5.12 ms time constant, 120 G sweep width and 6 scans. Furthermore, data reproducibility was checked by running ESR measurements repeatedly during four different days. ESR intensities were extracted

from peak-to-peak amplitude measurements between T1 and B2 peaks at g=2.0018 (Grün et al., 2008). The Equivalent Doses (D_E) were calculated with the Microcal Origin 8.5 software using a Single Saturating Exponential (SSE) (Fig. SM-2 in Supplementary Material) function (Yokoyama et al., 1985). The irradiated point at 320 Gy was systematically underestimated showing the samples did not receive the expected dose. So, we decided to subtract this point for all the samples allowing to avoid an overestimation of the D_E.

4.4. U-Th analyses

The dental tissues (enamel and a part of cement) were analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at *Musée de l'Homme* in Paris (France), using a Thermo-Electron iCAP-RQ quadripole mass spectrometer and following the procedure described in Douville et al. (2010). The chemical protocol is explained in Richard et al. (2015). The dentine tissues and the rest of cement samples were analyzed by a Multicollector Inductively Coupled Plasma Mass Spectrometry (MC-ICPMS) at *Nanjing Normal University* in Nanjing (China) following a protocol similar to that explained in Shao et al. (2019). The main steps consist in adding a ^{233}U-^{236}U-^{229}Th spike to the dissolved sample. U and Th are then separated and purified on UTEVA resin columns. Distilled and clean nitric and hydrochloric acids were used according to each step. The U and Th fractions are eventually pre-analyzed in order to prepare the adapted final dilutions for the best measurements, either on Q-ICPMS or MC-ICPMS. An uraninite (HU-1) standard with spike, in secular radioactive equilibrium, is analyzed regularly for instrument control.

The total blank chemistry values must be as low as possible and the internal standard sample values after application of the various corrections must be corrected to validate the ages. Ages were calculated using half-lives of 75,584 years for ^{230}Th and 245,620 years for ^{234}U (Cheng et al., 2013). All errors are reported at 95% confidence level.

4.5. Dose rate evaluation

The Dose Rate (D) was obtained from the sum of the α-, β-, γ- and cosmic-ray contributions. As teeth were handed over after the excavation campaigns and because most of the layers were excavated and disappeared, no *in situ* measurements of the natural radioactivity of the sediment could be made except for sample GE1802. This single *in situ* measurement was carried out using a NaI(Tl) probe connected to an Inspector 1000 multichannel analyser (Canberra) and the dose rate (115 ± 50 µGy/a) was calculated using the "threshold approach" (Mercier and Falguères, 2007). External ß contributions were assessed

from the radioelement content (U, Th and K) determined by high resolution low background gamma-ray spectrometry (HRGS) at the *Centro Nacional de Investigación sobre la Evolución Humana (CENIEH)* in Burgos (Spain). The cosmic dose rate was estimated according to the depth of the sediments, the thickness of the limestone roof, and taking into account the altitude and latitude using the formula of Prescott and Hutton (1994).

5. Results and discussion

ESR/U-series ages were calculated using the DATA software (Grün, 2009) except for GE1803 which was calculated using the US-ESR program allowing a slight uranium leaching in the tissues (Shao et al., 2014). Seven of the nine teeth analysed have yielded modelled ages and are presented in Table 1 and Fig 2.

Table 1. Approx. here

Equivalent Doses (D_E) are homogeneous and range between 27 ± 5 and 43 ± 5 Gy for samples from the GE-I test pit. The D_E obtained for the only sample from the GE-II test pit that could be analyzed is slightly higher (52 ± 4 Gy). The external dose rate due to the surrounding sediment is preponderant in the total Dose Rate (D): between 87% and 96% for GE-I teeth, reaching 98% for GE1808 in GE-II. ^{238}U content in enamel is very low and ranges between 0.013 ± 0.001 to 0.070 ± 0.002 ppm excepting for GE1804 and GE1806 which contain 5 to 10 times more. The U-content in dentine is higher, ranging from 0.313 ± 0.001 to 1.063 ± 0.001 ppm while cement is the tissue with the highest U-content, ranging from 0.735 ± 0.001 to 17.86 ± 0.63 ppm.

The difficulties to obtain reliable and accurate ages from the fossil teeth of the Galería de las Estatuas site come from two main reasons. Firstly, teeth have low U-content in enamel and the limits of U-series measurements by ICPQMS are reached. Although it was technically possible to calculate US-ESR ages, the obtained *p*-values show a large error range (Table 1). GE1804 probably acquired post-burial uranium very late, at the beginning of the Holocene period as suggested by positive *p*-values higher than seven obtained for the three tissues.

Secondly, the heterogeneity of the layers renders difficult the measurement and the evaluation of the external dose rate which represents more than 80% of the total Dose Rate and for some samples, almost 100%. For example, sample GE1801 (276 ± 21 ka) has provided an older age than expected and an external dose rate twice lower than for others

samples. The LU-5, where GE1801 was found, has been described as the result of the degradation of an underlying speleothem and shows a highly carbonated composition (Arsuaga et al., 2017). The difficulty of assessing the external dose rate in this highly carbonate layer coupled with the low U-content in the enamel could be likely explain this older age. It is difficult to further refine this age and it should be used with caution to make interpretations on this sequence.

The results obtained in the GE-I test pit suggest that the analyzed teeth are contemporaneous with MIS 5 ranging between 92 ± 6 ka and 104 ± 6 ka for LU-5 to LU-2. These ages are in accordance with the single-grain OSL ages suggesting that the sediment sequence and archaeological remains excavated in GE-I may have been accumulated 80-112 ka. In the GE-II test pit, the ESR/U-series age obtained (115 ± 18 ka) is slightly older than that proposed by single-grain TT-OSL which were deposited 70-79 ka.

6. Conclusions

The Galería de las Estatuas site is the first site within the Sierra de Atapuerca to provide Neanderthal human fossil in a well-established stratigraphic context (Pablos et al., 2019), as well as Neanderthal DNA extracted from sediments (Vernot et al., 2021). The single-grain OSL and TT-OSL ages obtained for the two test pits excavated since 2008 revealed an age for GE-I to approximately 80-112 ka and 70-79 ka por GE-II suggesting that the GE site is one of the oldest Neanderthal sites (MIS 5) in the Iberian Peninsula (Demuro et al., 2019).

Therefore, the importance of this site highlights the need to contrast the previous dates with others obtained with a different methodology in order to verify and refine the age of this site. In that regard, nine fossil teeth were sampled in GE to be dated by combined ESR/U-series dating method: seven teeth from GE-I and two teeth from GE-II.

In GE-I test pit, six out of the seven teeth analysed provided model ages. The results for GE-I suggest that teeth are contemporaneous with MIS 5 ranging between 92 and 104 ka for LU-5 to LU-2. The older age obtained for GE1801 (LU-5 from GE-I) should be used with caution for interpretations of this sequence. The heterogeneity of the layer and the high carbonate content rendering difficult the measurement of the external dose rate seems to explain the older age obtained.

In GE-II test pit, only one of the two teeth analysed has provided a model age. The sample GE1808 has provided an age of 115 ± 18 ka which is slightly older than the single-grain TT-OSL (86 ± 8 ka in LU-1 and 84 ± 7 ka in LU-2) ages obtained by Demuro et al.

(2019).

Most of the ESR/U-series ages obtained in this work are in agreement with the previous single-grain OSL and TT-OSL ages and confirm the Upper Pleistocene age of the Galería de las Estatuas site within late MIS 5 and early MIS 4.

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Author contributions

DM, CF and AIO designed the study; AGO and AA provided general and stratigraphic background about the site; DM and CF performed the *in situ* measurements and collected data in the site; DM, CF, OT, QS, AIO and FT carried out the geochronological analysis; JLA, JMBC, EC and DM provided scientific support and resources from PGC2018-093925-B-C31, PGC2018-093925-B-C32, PGC2018-093925-B-C33 and CAS18/00404; DM wrote the paper with inputs from the others.

References

Apellániz, J.M., Domingo Mena, A., 1987. Los materiales de superficie del santuario de la Galería del Sílex, in: Cuadernos de Arqueología de Deusto. Servicio de Publicaciones de la Universidad de Deusto, Bilbao, pp. 5–278.

Aranburu, A., Martínez-Pillado, V., García, F., Arsuaga, J.L., Alcázar, A., de Velasco, Bonmati, A., García, N., Gracia, A., Gracia, F., Gómez, A., Lira, J., Lorenzo, C., Martínez, I., Ortega, A.I., Pablos, A., Pantoja, A., Quam, R., Sala, N., 2012. La variabilidad de los rellenos endokársticos de la Galería de las Estatuas (Atapuerca, Burgos) y su caracterización paleoambiental, in: González Díez, A., Fernández-

Maroto, G., Doughty, M.W., Remondo, J., Bruschi, V.M., Bonachea, J., Díaz de Terán, J.R., Martínez-Cedrún, P., Rivas, V., Flor, G., Serrano, E., Gutiérrez Santolalla, F., Benito, G., Desir, G., López, J., Romero, M.A., Batalla, R., Gracia-Prieto, F.J., Flor-Blanco, G., Garzón, G., Cardenal, J., Vilaplana, J.M., Corominas, J., Díaz-Martínez, E., Jiménez-Sánchez, M., Guerra, J.C. (Eds.), Avances de la Geomorfología en España 2010–2012. Actas de la XII Reunión Nacional de Geomorfología (Santander, 17–20 septiembre de 2012. PUbliCan. Ediciones de la Universidad de Cantabria, D.L., Santander, p. 690.

Arnold, L.J., Demuro, M., Navazo, M., Benito-Calvo, A., Pérez-González, A., 2013. OSL dating of the Middle Palaeolithic Hotel California site, Sierra de Atapuerca, north-central Spain: OSL dating of the Middle Palaeolithic Hotel California site, Spain. Boreas 42, 285–305. https://doi.org/10.1111/j.1502-3885.2012.00262.x

Arsuaga, J.L., Gómez-Olivencia, A., Sala, N., Martínez-Pillado, V., Pablos, A., Bonmatí, A., Pantoja-Pérez, A., Lira-Garrido, J., Alcázar de Velasco, A., Ortega, A.I., Cuenca-Bescós, G., García, N., Aranburu, A., Ruiz-Zapata, B., José Gil-García, M., Rodríguez-Álvarez, X.P., Ollé, A., Mosquera, M., 2017. Evidence of paleoecological changes and Mousterian occupations at the Galería de las Estatuas site, Sierra de Atapuerca, northern Iberian plateau, Spain. Quaternary Research 88, 345–367. https://doi.org/10.1017/qua.2017.46

Carbonell, E., Huguet, R., Cáceres, I., Lorenzo, C., Mosquera, M., Ollé, A., Rodriguez, X.P., Saladié, P., Vergès, J.M., García, P., Rosell, J., Vallverdú, J., Carretero, J.M., Navazo, M., Ortega, A.I., Martinón-Torres, M., Muro, J.I., Allué, E., Aranburu, A., Canals, A., Carrancho, Á., Castilla, M., Expósito, I., Fontanals, M., Francés-Negro, M., Galindo-Pellicena, M.Á., García-Antón, M.D., García, N., Gracia, F., Gómez-Merino, G., García, R., Iriarte, E., de Lombera-Hermida, A., López-Polín, L., Lozano, M., van der Made, J., Martínez, I., Mateos, A., Pérez-Romero, A., Poza-Rey, E., Quam, R., Rodríguez, A., Rodríguez, J., Rodríguez, L., Santos, E., Terradillos, M., Bermúdez de Castro, J.M., Arsuaga, J.L., 2014. Sierra de Atapuerca archaeological sites, in: Sala, R., Carbonell i Roura, E., Bermúdez de Castro, J.M., Arsuaga, J.L. (Eds.), Pleistocene and Holocene hunter-gatherers in Iberia and the Gibraltar strait: the current archaeological record. Fundación Atapuerca - Universidad de Burgos, Servicio de publicaciones, pp. 534–560.

Cheng, H., Lawrence Edwards, R., Shen, C.-C., Polyak, V.J., Asmerom, Y., Woodhead, J., Hellstrom, J., Wang, Y., Kong, X., Spötl, C., Wang, X., Calvin Alexander, E., 2013. Improvements in 230Th dating, 230Th and 234U half-life values, and U–Th isotopic measurements by multi-collector inductively coupled plasma mass spectrometry. Earth and Planetary Science Letters 371–372, 82–91. https://doi.org/10.1016/j.epsl.2013.04.006

Demuro, M., Arnold, L.J., Aranburu, A., Gómez-Olivencia, A., Arsuaga, J.-L., 2019. Single-grain OSL dating of the Middle Palaeolithic site of Galería de las Estatuas, Atapuerca (Burgos, Spain). Quaternary Geochronology 49, 254–261. https://doi.org/10.1016/j.quageo.2018.02.006

Douville, E., Sallé, E., Frank, N., Eisele, M., Pons-Branchu, E., Ayrault, S., 2010. Rapid and accurate U–Th dating of ancient carbonates using inductively coupled plasma-quadrupole mass spectrometry. Chemical Geology 272, 1–11. https://doi.org/10.1016/j.chemgeo.2010.01.007

Grün, R., 2009. The DATA program for the calculation of ESR age estimates on tooth enamel. Quaternary Geochronology 4, 231–232. https://doi.org/10.1016/j.quageo.2008.12.005

Grün, R., Joannes-Boyau, R., Stringer, C., 2008. Two types of CO2− radicals threaten the

fundamentals of ESR dating of tooth enamel. Quaternary Geochronology 3, 150–172. https://doi.org/10.1016/j.quageo.2007.09.004

Martínez-Pillado, V., Aranburu, A., Arsuaga, J., Ruiz-Zapata, B., Gil-García, M., Stoll, H., Yusta, I., Iriarte, E., Carretero, J., Edwards, R., Cheng, H., 2014. Upper Pleistocene and Holocene palaeoenvironmental records in Cueva Mayor karst (Atapuerca, Spain) from different proxies: speleothem crystal fabrics, palynology, and archaeology. International Journal of Speleology 43, 1–14. https://doi.org/10.5038/1827-806X.43.1.1

Mercier, N., Falguères, C., 2007. Field gamma dose-rate measurement with a NaI(Tl) detector: re-evaluation of the "threshold" technique. Ancient TL 25, 1–4.

Mosquera, M., Olle, A., Pérez-González, A., Rodríguez, X.P., Vaquero, M., Verges, J.M., Carbonell, E., 2007. Valle de las Orquídeas: an Upper Pleistocene open-air site at Sierra de Atapuerca (Burgos). Trabajos de Prehistoria 64, 143–155. https://doi.org/10.3989/tp.2007.v64.i2.113

Navazo, M., Alonso-Alcalde, R., Benito-Calvo, A., Díez, J.C., Pérez-González, A., Carbonell, E., 2011. Hundidero: MIS 4 open air Neanderthal occupations in Sierra de Atapuerca. Archaeology, Ethnology and Anthropology of Eurasia 39, 29–41. https://doi.org/10.1016/j.aeae.2012.02.004

Navazo, M., Carbonell, E., 2014. Neanderthal settlement patterns during MIS 4–3 in Sierra de Atapuerca (Burgos, Spain). Quaternary International 331, 267–277. https://doi.org/10.1016/j.quaint.2014.03.032

Ortega, A.I., 2009. La evolución geomorfológica del karst de la Sierra de Atapuerca (Burgos) y su relación con los yacimientos pleistocenos que contiene (PhD). Universidad de Burgos, Burgos.

Ortega, A.I., Benito-Calvo, A., Pérez-González, A., Martín-Merino, M.A., Pérez-Martínez, R., Parés, J.M., Aramburu, A., Arsuaga, J.L., Bermúdez de Castro, J.M., Carbonell, E., 2013. Evolution of multilevel caves in the Sierra de Atapuerca (Burgos, Spain) and its relation to human occupation. Geomorphology 196, 122–137. https://doi.org/10.1016/j.geomorph.2012.05.031

Pablos, A., Gómez-Olivencia, A., Arsuaga, J.L., 2019. A Neandertal foot phalanx from the Galería de las Estatuas site (Sierra de Atapuerca, Spain). American Journal of Physical Anthropology 168, 222–228. https://doi.org/10.1002/ajpa.23729

Prescott, J.R., Hutton, J.T., 1994. Cosmic ray contributions to dose rates for luminescence and ESR dating: Large depths and long-term time variations. Radiation Measurements 23, 497–500. https://doi.org/10.1016/1350-4487(94)90086-8

Richard, M., Falguères, C., Pons-Branchu, E., Bahain, J.-J., Voinchet, P., Lebon, M., Valladas, H., Dolo, J.-M., Puaud, S., Rué, M., Daujeard, C., Moncel, M.-H., Raynal, J.-P., 2015. Contribution of ESR/U-series dating to the chronology of late Middle Palaeolithic sites in the middle Rhône valley, southeastern France. Quaternary Geochronology 30, 529–534. https://doi.org/10.1016/j.quageo.2015.06.002

Santamaría, M., Navazo, M., Benito-Calvo, A., Alonso, R., López, G.I., Carbonell, E., 2021. Atapuerca Neanderthal landscape at Fuente Mudarra site in Burgos, Spain, during Marine Isotope Stages 5–3. Quaternary Research 99, 248–269. https://doi.org/10.1017/qua.2020.65

Shao, Q., Bahain, J.-J., Dolo, J.-M., Falguères, C., 2014. Monte Carlo approach to calculate US-ESR age and age uncertainty for tooth enamel. Quaternary Geochronology 22, 99–106. https://doi.org/10.1016/j.quageo.2014.03.003

Shao, Q.-F., Li, C.-H., Huang, M.-J., Liao, Z.-B., Arps, J., Huang, C.-Y., Chou, Y.-C., Kong, X.-G., 2019. Interactive programs of MC-ICPMS data processing for 230Th/U geochronology. Quaternary Geochronology 51, 43–52.

https://doi.org/10.1016/j.quageo.2019.01.004
Vernot, B., Zavala, E.I., Gómez-Olivencia, A., Jacobs, Z., Slon, V., Mafessoni, F., Romagné, F., Pearson, A., Petr, M., Sala, N., Pablos, A., Aranburu, A., de Castro, J.M.B., Carbonell, E., Li, B., Krajcarz, M.T., Krivoshapkin, A.I., Kolobova, K.A., Kozlikin, M.B., Shunkov, M.V., Derevianko, A.P., Viola, B., Grote, S., Essel, E., Herráez, D.L., Nagel, S., Nickel, B., Richter, J., Schmidt, A., Peter, B., Kelso, J., Roberts, R.G., Arsuaga, J.-L., Meyer, M., 2021. Unearthing Neanderthal population history using nuclear and mitochondrial DNA from cave sediments. Science 372, eabf1667. https://doi.org/10.1126/science.abf1667
Yokoyama, Y., Falguères, C., Quaegebeur, J.P, 1985. ESR dating of quartz from Quaternary sediments: first attempt. Nuclear Tracks and Radiation Measurements 10, 921–928.

Aranburu, A., Martínez-Pillado, V., García, F., Arsuaga, J.L., Alcázar, A., de Velasco, Bonmati, A., García, N., Gracia, A., Gracia, F., Gómez, A., Lira, J., Lorenzo, C., Martínez, I., Ortega, A.I., Pablos, A., Pantoja, A., Quam, R., Sala, N., 2012. La variabilidad de los rellenos endokársticos de la Galería de las Estatuas (Atapuerca, Burgos) y su caracterización paleoambiental, in: González Díez, A., Fernández-Maroto, G., Doughty, M.W., Remondo, J., Bruschi, V.M., Bonachea, J., Díaz de Terán, J.R., Martínez-Cedrún, P., Rivas, V., Flor, G., Serrano, E., Gutiérrez Santolalla, F., Benito, G., Desir, G., López, J., Romero, M.A., Batalla, R., Gracia-Prieto, F.J., Flor-Blanco, G., Garzón, G., Cardenal, J., Vilaplana, J.M., Corominas, J., Díaz-Martínez, E., Jiménez-Sánchez, M., Guerra, J.C. (Eds.), Avances de la Geomorfología en España 2010–2012. Actas de la XII Reunión Nacional de Geomorfología (Santander, 17–20 septiembre de 2012. PUbliCan. Ediciones de la Universidad de Cantabria, D.L., Santander, p. 690.
Arnold, L.J., Demuro, M., Navazo, M., Benito-Calvo, A., Pérez-González, A., 2013. OSL dating of the Middle Palaeolithic Hotel California site, Sierra de Atapuerca, north-central Spain: OSL dating of the Middle Palaeolithic Hotel California site, Spain. Boreas 42, 285–305. https://doi.org/10.1111/j.1502-3885.2012.00262.x
Arsuaga, J.L., Gómez-Olivencia, A., Sala, N., Martínez-Pillado, V., Pablos, A., Bonmatí, A., Pantoja-Pérez, A., Lira-Garrido, J., Alcázar de Velasco, A., Ortega, A.I., Cuenca-Bescós, G., García, N., Aranburu, A., Ruiz-Zapata, B., José Gil-García, M., Rodríguez-Álvarez, X.P., Ollé, A., Mosquera, M., 2017. Evidence of paleoecological changes and Mousterian occupations at the Galería de las Estatuas site, Sierra de Atapuerca, northern Iberian plateau, Spain. Quaternary Research 88, 345–367. https://doi.org/10.1017/qua.2017.46
Carbonell, E., Huguet, R., Cáceres, I., Lorenzo, C., Mosquera, M., Ollé, A., Rodriguez, X.P., Saladié, P., Vergès, J.M., García, P., Rosell, J., Vallverdú, J., Carretero, J.M., Navazo, M., Ortega, A.I., Martinón-Torres, M., Muro, J.I., Allué, E., Aranburu, A., Canals, A., Carrancho, Á., Castilla, M., Expósito, I., Fontanals, M., Francés-Negro, M., Galindo-Pellicena, M.Á., García-Antón, M.D., García, N., Gracia, F., Gómez-Merino, G., García, R., Iriarte, E., de Lombera-Hermida, A., López-Polín, L., Lozano, M., van der Made, J., Martínez, I., Mateos, A., Pérez-Romero, A., Poza-Rey, E., Quam, R., Rodríguez, A., Rodríguez, J., Rodríguez, L., Santos, E., Terradillos, M., Bermúdez de Castro, J.M., Arsuaga, J.L., 2014. Sierra de Atapuerca archaeological sites, in: Sala, R., Carbonell i Roura, E., Bermúdez de Castro, J.M., Arsuaga, J.L. (Eds.), Pleistocene and Holocene hunter-gatherers in Iberia and the Gibraltar strait: the current archaeological record. Fundación Atapuerca - Universidad de Burgos, Servicio de publicaciones, pp. 534–560.
Cheng, H., Lawrence Edwards, R., Shen, C.-C., Polyak, V.J., Asmerom, Y., Woodhead, J.,

Hellstrom, J., Wang, Y., Kong, X., Spötl, C., Wang, X., Calvin Alexander, E., 2013. Improvements in 230Th dating, 230Th and 234U half-life values, and U–Th isotopic measurements by multi-collector inductively coupled plasma mass spectrometry. Earth and Planetary Science Letters 371–372, 82–91. https://doi.org/10.1016/j.epsl.2013.04.006

Demuro, M., Arnold, L.J., Aranburu, A., Gómez-Olivencia, A., Arsuaga, J.-L., 2019. Single-grain OSL dating of the Middle Palaeolithic site of Galería de las Estatuas, Atapuerca (Burgos, Spain). Quaternary Geochronology 49, 254–261. https://doi.org/10.1016/j.quageo.2018.02.006

Douville, E., Sallé, E., Frank, N., Eisele, M., Pons-Branchu, E., Ayrault, S., 2010. Rapid and accurate U–Th dating of ancient carbonates using inductively coupled plasma-quadrupole mass spectrometry. Chemical Geology 272, 1–11. https://doi.org/10.1016/j.chemgeo.2010.01.007

Grün, R., 2009. The DATA program for the calculation of ESR age estimates on tooth enamel. Quaternary Geochronology 4, 231–232. https://doi.org/10.1016/j.quageo.2008.12.005

Grün, R., Joannes-Boyau, R., Stringer, C., 2008. Two types of CO2− radicals threaten the fundamentals of ESR dating of tooth enamel. Quaternary Geochronology 3, 150–172. https://doi.org/10.1016/j.quageo.2007.09.004

Martínez-Pillado, V., Aranburu, A., Arsuaga, J., Ruiz-Zapata, B., Gil-García, M., Stoll, H., Yusta, I., Iriarte, E., Carretero, J., Edwards, R., Cheng, H., 2014. Upper Pleistocene and Holocene palaeoenvironmental records in Cueva Mayor karst (Atapuerca, Spain) from different proxies: speleothem crystal fabrics, palynology, and archaeology. International Journal of Speleology 43, 1–14. https://doi.org/10.5038/1827-806X.43.1.1

Mercier, N., Falguères, C., 2007. Field gamma dose-rate measurement with a NaI(Tl) detector: re-evaluation of the “threshold” technique. Ancient TL 25, 1–4.

Mosquera, M., Olle, A., Pérez-González, A., Rodríguez, X.P., Vaquero, M., Verges, J.M., Carbonell, E., 2007. Valle de las Orquídeas: an Upper Pleistocene open-air site at Sierra de Atapuerca (Burgos). Trabajos de Prehistoria 64, 143–155. https://doi.org/10.3989/tp.2007.v64.i2.113

Navazo, M., Alonso-Alcalde, R., Benito-Calvo, A., Díez, J.C., Pérez-González, A., Carbonell, E., 2011. Hundidero: MIS 4 open air Neanderthal occupations in Sierra de Atapuerca. Archaeology, Ethnology and Anthropology of Eurasia 39, 29–41. https://doi.org/10.1016/j.aeae.2012.02.004

Navazo, M., Carbonell, E., 2014. Neanderthal settlement patterns during MIS 4–3 in Sierra de Atapuerca (Burgos, Spain). Quaternary International 331, 267–277. https://doi.org/10.1016/j.quaint.2014.03.032

Ortega, A.I., 2009. La evolución geomorfológica del karst de la Sierra de Atapuerca (Burgos) y su relación con los yacimientos pleistocenos que contiene (PhD). Universidad de Burgos, Burgos.

Ortega, A.I., Benito-Calvo, A., Pérez-González, A., Martín-Merino, M.A., Pérez-Martínez, R., Parés, J.M., Aramburu, A., Arsuaga, J.L., Bermúdez de Castro, J.M., Carbonell, E., 2013. Evolution of multilevel caves in the Sierra de Atapuerca (Burgos, Spain) and its relation to human occupation. Geomorphology 196, 122–137. https://doi.org/10.1016/j.geomorph.2012.05.031

Pablos, A., Gómez-Olivencia, A., Arsuaga, J.L., 2019. A Neandertal foot phalanx from the Galería de las Estatuas site (Sierra de Atapuerca, Spain). American Journal of Physical Anthropology 168, 222–228. https://doi.org/10.1002/ajpa.23729

Prescott, J.R., Hutton, J.T., 1994. Cosmic ray contributions to dose rates for luminescence

and ESR dating: Large depths and long-term time variations. Radiation Measurements 23, 497–500. https://doi.org/10.1016/1350-4487(94)90086-8

Richard, M., Falguères, C., Pons-Branchu, E., Bahain, J.-J., Voinchet, P., Lebon, M., Valladas, H., Dolo, J.-M., Puaud, S., Rué, M., Daujeard, C., Moncel, M.-H., Raynal, J.-P., 2015. Contribution of ESR/U-series dating to the chronology of late Middle Palaeolithic sites in the middle Rhône valley, southeastern France. Quaternary Geochronology 30, 529–534. https://doi.org/10.1016/j.quageo.2015.06.002

Santamaría, M., Navazo, M., Benito-Calvo, A., Alonso, R., López, G.I., Carbonell, E., 2021. Atapuerca Neanderthal landscape at Fuente Mudarra site in Burgos, Spain, during Marine Isotope Stages 5–3. Quaternary Research 99, 248–269. https://doi.org/10.1017/qua.2020.65

Shao, Q., Bahain, J.-J., Dolo, J.-M., Falguères, C., 2014. Monte Carlo approach to calculate US-ESR age and age uncertainty for tooth enamel. Quaternary Geochronology 22, 99–106. https://doi.org/10.1016/j.quageo.2014.03.003

Shao, Q.-F., Li, C.-H., Huang, M.-J., Liao, Z.-B., Arps, J., Huang, C.-Y., Chou, Y.-C., Kong, X.-G., 2019. Interactive programs of MC-ICPMS data processing for 230Th/U geochronology. Quaternary Geochronology 51, 43–52. https://doi.org/10.1016/j.quageo.2019.01.004

Vernot, B., Zavala, E.I., Gómez-Olivencia, A., Jacobs, Z., Slon, V., Mafessoni, F., Romagné, F., Pearson, A., Petr, M., Sala, N., Pablos, A., Aranburu, A., de Castro, J.M.B., Carbonell, E., Li, B., Krajcarz, M.T., Krivoshapkin, A.I., Kolobova, K.A., Kozlikin, M.B., Shunkov, M.V., Derevianko, A.P., Viola, B., Grote, S., Essel, E., Herráez, D.L., Nagel, S., Nickel, B., Richter, J., Schmidt, A., Peter, B., Kelso, J., Roberts, R.G., Arsuaga, J.-L., Meyer, M., 2021. Unearthing Neanderthal population history using nuclear and mitochondrial DNA from cave sediments. Science 372, eabf1667. https://doi.org/10.1126/science.abf1667

Yokoyama, Y., Falguères, C., Quaegebeur, J.P, 1985. ESR dating of quartz from Quaternary sediments: first attempt. Nuclear Tracks and Radiation Measurements 10, 921–928.

FIGURES

Fig. 1. (A) Location and plan of the Sierra de Atapuerca cave systems showing the main archaeological sites (modified from G.E. Edelweiss) (B) Detailed view and cross-section of the end of the Galería de las Estatuas and the position of the test pits GE-I and GE-II *(modified from* Ortega (2009).

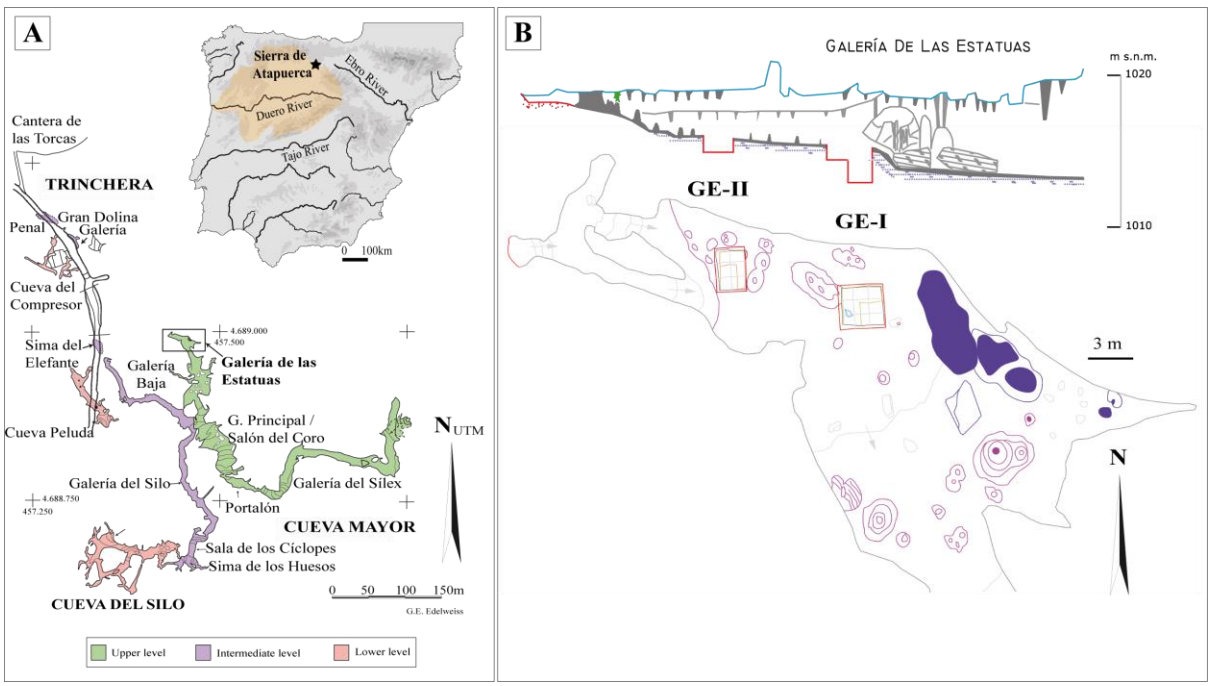
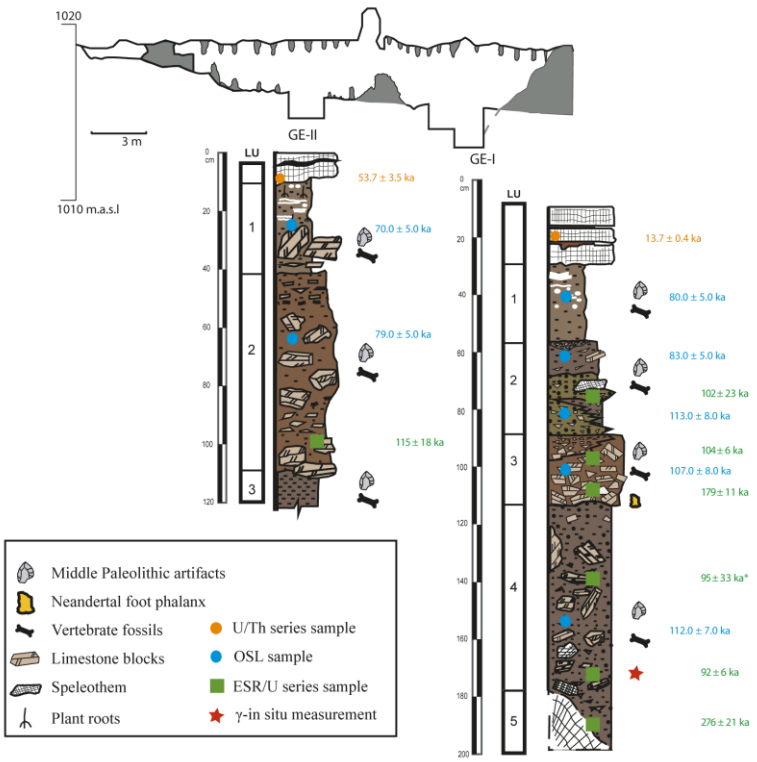


Fig 2. Location of the ESR/U-series ages in relationship with the OSL and U-Th ages in the stratigraphical profiles from GE-I and GE-II *(modified from* Vernot et al. (2021).



TABLES

Table 1. Total dose rates (D), Equivalent Doses (D_E), U content, *p*-values and ages determined for teeth from Galería de las Estatuas site. (* as U dentine was not measured by U-series, a value corresponding to that of the other dentines coming from the closest horizon was chosen and a modelled age was calculated). LU: lithostratigraphic unit; D_{ext}: external dose; D_{int}: internal dose.

Test pit	Samples	LU	U content (ppm)			D_E (Gy)	D_{ext} (β + γ) (µGy/a)	D_{int} (α+β) (µGy/a)	D (µGy/a)	*p*-value			ESR/U-series ages (ka)
			Enamel	Dentine	Cement					enamel	dentine	cement	
GE-I	GE1806*	2	0,261 ± 0,001	1	17,86 ± 0,63	43 ± 5	454 ± 68	115 ± 73	569 ± 125	-0,92 ± 0,19	-0,75 ± 0,23	-0,75 ± 0,23	**102 ±23**
	GE1804	3	0,273 ± 0,008	1,063 ± 0,001	9,133 ± 0,002	29 ± 2	317 ± 60	3 ± 2	320 ± 60	9,79 ± 0,72	9,00 ± 0,67	7,04 ± 0,55	**104 ± 6**
	GE1805	3	0,066 ± 0,002	0,850 ± 0,001	3,836 ± 0,002	36 ± 3	220 ± 61	6 ± 3	226 ± 61	1,18 ± 0,18	1,43 ± 0,20	1,18 ± 0,18	**179 ± 11**
	GE1803*	4	0,070 ± 0,002	0,4	4,344 ± 0,13	31 ± 4	317 ± 60	64 ± 50	381 ± 134	-0,17 ± 0,57	-0,02 ± 0,05	-0,02 ± 0,05	**95 ± 33**
	GE1802	4	0,013 ± 0,001	0,313 ± 0,001	1,236 ± 0,04	27 ± 5	317 ± 60	3 ± 3	320 ± 63	-0,89 ± 0,06	0,51 ± 0,15	0,29 ± 0,14	**92 ± 6**
	GE1801	5	0,032 ± 0,001	0,423 ± 0,001	4,167 ±0,001	31 ± 5	110 ± 41	0	110 ± 41	-0,88 ± 0,04	6,15 ± 0,59	0,58 ± 0,16	**276 ± 21**
GE-II	GE1808	2	0,040 ± 0,001	0,856 ± 0,001	0,735 ± 0,001	52 ± 4	508 ± 72	6 ± 3	514 ± 84	-0,19 ± 0,24	1,59 ± 0,53	0,92 ± 0,42	**115 ± 18**