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New electron spin resonance (ESR) ages from Geißenklösterle Cave: A chronological study on Middle and Early Upper Paleolithic layers

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**Abstract** 

Geißenklösterle Cave (Germany) is one of the most important Paleolithic sites in Europe, as it is characterized by human occupation during the Middle and Early Upper Paleolithic. Aurignacian layers prior to 37–38 ka cal BP feature both musical and figurative art objects that are linked to the early arrival in Europe of *Homo sapiens*. Middle Paleolithic layers yielded lithic artifacts attributed to *Homo neanderthalensis*. Since human occupation at the site is attributed to both Neanderthals and modern humans, chronology is essential to clarify the issues of Neanderthal disappearance, modern humans expansion in Europe, and the origin of the Aurignacian in Western Europe. Electron spin resonance (ESR) dating was performed on fossil tooth enamel collected from the Middle Paleolithic layers, which are beyond the radiocarbon dating range, and from the nearly sterile 'transitional' geological horizon (GH) 17 and the lower Aurignacian deposits, to cross-check ESR ages with previous radiocarbon, thermoluminescence and ESR age results. The Middle Paleolithic layers were 43 dated between 94  $\pm$  10 ka (GH 21) and 55  $\pm$  6 ka (GH 18) by ESR on tooth enamel. Mean 44 ages for GH 17, at  $46 \pm 3$  ka, and for the lower Aurignacian layers, at  $37 \pm 3$  ka, are in agreement with previous dating results, thus supporting the reliability of ESR chronology for the base of the sequence where dating comparisons are not possible. These results suggest that Neanderthals occupied the site from Marine Isotope Stage (MIS) 5 to the second half of MIS 3 and confirm the antiquity of early Aurignacian deposits. The presence of an almost sterile layer that separates Middle and Upper Paleolithic occupations could be related to the abandonment of the site by Neanderthals, probably during Heinrich Stadial 5 (ca. 49–47 ka), thus before the arrival of *H. sapiens* in the area around 42 ka cal BP. These dates for the Middle Paleolithic of the Swabian Jura represent an important contribution to the prehistory of the region, where nearly all of the excavations were conducted decades ago and prior to the development of reliable radiometric dating beyond the range of radiocarbon.

#### **1. Introduction**

#### *1.1. Dating the end of the Middle Paleolithic and the beginning of the Upper Paleolithic*

The Middle to Upper Paleolithic transition has been the subject of numerous studies and has been a topic of lively debate for the past thirty years (e.g., White, 1982; Mellars, 1999; Conard, 2006; Villa and Roebroeks, 2014). Different hypotheses have been proposed to explain the disappearance of Neanderthals (*Homo neanderthalensis*) and the arrival and expansion of anatomically modern humans (*Homo sapiens*) in Eurasia during the Late Pleistocene (e.g., Mellars, 2004; Hockett and Haws, 2005; Conard et al., 2006; Verpoorte, 2006; Müller et al., 2011; Harvati-Papatheodorou, 2013). The cultural interactions between these two species are still debated and two main hypotheses are often invoked to explain the changes observed in the technical behavior during this crucial period. The presence of 'transitional industries' from around 45 ka to 30 ka (e.g., Douka et al., 2014), or as early as 48 ka (Richter et al., 2009) across Europe has been interpreted either as the result of Neanderthals and modern humans 'cohabitation', which led to cultural exchanges (the acculturation hypothesis; e.g., Hublin et al., 1996; Mellars, 2005) or as an endogenous development attributed to Neanderthals (the multiple-species model, e.g., d'Errico et al., 1998; d'Errico, 2003; Zilhão et al., 2006). The emergence of the Aurignacian, which broadly coincides with the first known *H. sapiens* in Western Europe (Higham et al., 2011, 2012) also raises questions about the development of both figurative and musical art out of Africa (e.g., Conard, 2010, 2015). During the last twenty years, the advances in dating methods have been

crucial for the construction of hypotheses regarding the beginning of the Upper Paleolithic, 77 particularly radiocarbon  $(^{14}C; e.g.,$  Higham et al., 2009). Conard and Bolus (2003, 2008) 78 published  $^{14}C$  ages for Aurignacian deposits from several sites in the Swabian Jura (e.g., Hohlenstein-Stadel, Hohle Fels, Geißenklösterle). Spreading from around 42 to 30 ka cal BP, these Aurignacian layers document one of the earliest evidence of artistic behavior in Western Europe (Conard, 2003, 2009). This chronology, later confirmed and improved by Higham et al. (2012), was essential to the construction of the 'Kulturpumpe' hypothesis (Conard and Bolus, 2003). According to this hypothesis, the Swabian Jura was a key center of cultural innovations for both the Aurignacian and the Gravettian, and many of the innovations from the region subsequently spread to other parts of Europe. In the 'weak' version of the Kulturpumpe hypothesis, the Swabian Jura was one of multiple regions for producing important innovations. In the 'strong' version of the hypothesis, the region would be the original source of key innovations of the Aurignacian including, but not limited to personal ornaments with a cultural dictated, three-dimensional form, figurative art, therianthropic imagery and musical instruments (Conard and Bolus, 2003).

During the last decade, radiocarbon dating played a key role in establishing well-defined transitional, late Middle and Upper Paleolithic chronologies. Methodological improvements allowed the revision of 'late chronologies', whose ages were potentially affected by modern carbon contaminations (e.g., Higham et al., 2009; Higham, 2011; Wood et al., 2013). Simultaneously, the radiocarbon calibration curve was extended to 50 ka cal BP (Reimer et al., 2013). However, for older Middle Paleolithic deposits, the chronology is still incomplete and imprecise. This is the case at Geißenklösterle Cave, which is a key site for the study of human behavior during the Late Pleistocene. The stratigraphy contains Middle and Upper Paleolithic occupations, which are separated by an occupational hiatus (Hahn, 1988). The upper part of the stratigraphic sequence, especially the Aurignacian layers, is well dated using radiocarbon (Conard and Bolus, 2003, 2008; Higham et al., 2012), which confirms 102 thermoluminescence (TL) ages (Richter et al., 2000).

The present work mainly focuses on the dating of the Middle Paleolithic deposits using the ESR dating method on fossil tooth enamel, in order to provide chronological constraints for the most ancient Neanderthal occupations in this cave. Samples from the 'occupational hiatus' GH 17, located between the Middle and Upper Paleolithic layers, and from the early Aurignacian GHs 15 and 16, were also analyzed with the aim of comparing 108 ESR ages with the published  ${}^{14}C$  and TL results.

*1.2. Geißenklösterle Cave* 

Geißenklösterle Cave is located near Blaubeuren in the Swabian Jura (Baden-Württemberg, Germany), a region of low mountains in southwest Germany (Fig. 1). The cave entrance faces the Ach Valley and opens above the river Ach, a Danube tributary. The region is famous for some of the most ancient examples of both musical and figurative art, attributed to *H. sapiens* (e.g., Hahn, 1971, 1988; Conard, 2003, 2009; Conard et al., 2009; Kind et al., 2014). Middle Paleolithic occupations, attributed to Neanderthals (Conard et al., 2012), are also numerous in the Swabian Jura. At Geißenklösterle Cave, it has been suggested that these two human species successively occupied the cave, making this site and its chronology particularly important for the debate on the origins of the Aurignacian in Western Europe (Sauvet et al., 2008; Hublin, 2012; Floss, 2017).

The stratigraphy is comprised by 22 geological horizons (GH) which are grouped in several archaeological horizons (AH), documenting Middle Paleolithic to Mesolithic occupations (Fig. 2). The AHs have been defined by stratigraphic and technocultural criteria. AH IV–VIII correspond to the Middle Paleolithic sequence of the site, while AH II represents the upper Aurignacian and AH III the lower Aurignacian deposits. The many subunits of AH I

include Mesolithic, Magdalenian and Gravettian occupations. Geological horizons have been distinguished according to sedimentology and numbered 1 to 23 (Hahn, 1988; Miller, 2015). In our study, we focused on Middle Paleolithic layers (GH 21-AH VII and GH 18-AH IV), 'transitional' GH 17 (AH IIIc, almost sterile, interpreted as an occupational hiatus) and early Aurignacian layers (GHs 15 and 16, AH III).

The Aurignacian layers (AHs II and III), rich in archaeological material, are characterized by the presence of lithic tools such as carinated and nosed endscrapers, large blades with lateral Aurignacian retouch, busked and carinated burins, as well as artifacts made of ivory, bone and antler (Fig. 3). Ornaments were also documented, such as perforated fox canines or ivory pendants in AH III and a pendant made of antler in AH II (Hahn, 1988). The discovery of flutes in AH II (Hahn, 1988), dated to around 40 ka (Higham et al., 2012), documents, together with the flutes from Hohle Fels and Vogelherd, the earliest known tradition of musical instruments (Conard et al., 2009). Ivory figurines depicting humans, mammoths, bears and bisons were also uncovered in AH II (Hahn, 1988). However, in the Middle Paleolithic layers, the density of material is significantly lower. The lithic assemblages are characterized by Levallois reduction and small numbers of side scrapers and retouched flakes (Fig. 4; Conard et al., 2006).

The faunal remains documented in the Middle Paleolithic and early Aurignacian layers are associated to a cold climate, related to a 'mammoth steppe environment' (Guthrie, 1990), as attested by the presence of reindeer (*Rangifer tarandus*), mammoth (*Mammuthus primigenius*), woolly rhino (*Coelodonta antiquitatis*) and wild horse (*Equus ferus*). Given the abundant presence of cave bear (*Ursus spelaeus*), it seems that Geißenklösterle Cave was used as a hibernation den (Münzel and Conard, 2004). The high concentration in mammoth remains is likely linked to the use of ivory by humans during the Aurignacian period, as documented by the presence of ivory points (Münzel, 2001). The density of faunal remains is much lower in the Middle Paleolithic layers, perhaps in relation with the size of the human groups that occupied the cave. The population density and the occupation intensity would have been lower during the Middle Paleolithic than during the Aurignacian period (Münzel and Conard, 2004; Conard et al., 2012).

The exceptional archaeological sequence of Geißenklösterle was the subject of several dating studies, mainly focused on the Upper Paleolithic layers, which are of great importance with regard to the emergence and development of the Aurignacian culture. Hypotheses such as the 'Kulturpumpe' and the 'Danube corridor' (according to which the Danube Valley would have been the main migration route between Southeastern Europe and the Swabian Jura; Conard and Bolus, 2003) are based on the chronology established for the lower Aurignacian deposits at Geißenklösterle. Chronological data thus played a key role in the interpretation of human dynamics during the Late Pleistocene and radiocarbon dating was consequently widely applied to samples from Upper Palaeolithic layers. Richter et al. (2000) published TL and ESR ages obtained on burnt flints and fossil tooth, respectively. The first set 165 of ages provided a mean TL age of  $40.2 \pm 1.5$  ka for the lower Aurignacian of AH III, coinciding with the Hengelo Interstadial. Four ESR ages were obtained on the late Middle 167 Paleolithic layer GH 18 (AH IV), ranging from  $53 \pm 7$  ka to  $36 \pm 5$  ka (mean age = 43.3  $\pm$  4.0 ka). They suggest that the last Neanderthals occupied the site between the end of MIS 4 and the beginning of MIS 3.

170 A large number of  ${}^{14}C$  ages were obtained on bones from several laboratories, mostly from the Aurignacian horizons (AHs II and III), synthesized in Conard and Bolus (2008). 172 They show wide variation, ranging from around 41 to 33 ka cal BP. Only a few  $^{14}C$  ages have been reported from the Middle Paleolithic levels, ranging from around 45 (AH VIII) to 37 ka cal BP (AH IV). It has been argued by Conard (2002) and Conard and Bolus (2003) that 175 important variations in atmospheric  $^{14}$ C concentration between 50 ka and 30 ka were the cause for some of the younger radiocarbon ages, based on the data obtained in North Atlantic planktonic foraminifera (Voelker et al., 2000), Japanese varves (Kitagawa and van der Plicht, 1998) and stalagmites from the Bahamas (Beck et al., 2001). Called the 'Middle Paleolithic Dating Anomaly', this phenomenon supposedly was responsible for interpretations of Middle and Upper Paleolithic occupations overlapping in time, and thus exaggerating the 'coexistence effect' between late Neanderthals and early *H. sapiens*. Higham et al. (2012) 182 published the calibrated data from Conard and Bolus (2008) as well as new  $^{14}C$ determinations of ultrafiltered collagen. Their results confirmed the antiquity of the Aurignacian layers, as suggested by Richter et al. (2000), which were deposited as early as 40 ka cal BP. More specifically, Higham et al. (2012) used Bayesian modelling to date the start of the early Aurignacian between 43 and 42 ka cal BP. However, regarding the transitional horizon (GH 17) and the Middle Paleolithic layers, only a few ages have been reported. Taking into account available data for the Middle to Upper Paleolithic levels, the results indicate that the early Aurignacian deposits and the almost sterile GH 17 are older than 40 ka cal BP, and the Middle Paleolithic layers range between around 52 ka cal BP (AH VII) and 35 ka cal BP (AH IV; Conard and Bolus, 2003, 2008; Higham et al., 2012).

**2. Materials and methods** 

#### *2.1. Sampling and sample preparation*

Herbivore tooth samples (*n* = 13) were collected during several excavation campaigns, between 1978 and 2002 (Table 1; Supplementary Online Material [SOM] Fig. S1). The sediment matrix that embedded the teeth was therefore not preserved for radioelement analysis. Thus, a sediment sample from each dated unit—except GH 16, which had been entirely excavated—was collected on site in 2013 (Fig. 5).

The enamel, dentine and cement (in the case of equid teeth) were separated 201 mechanically and cleaned with an electric drill. At least 30 to 40  $\mu$ m was removed from each side of the enamel to exclude the volume irradiated by alpha particles contained in sediment, 203 cement or dentine. After grinding, the enamel was sieved and the 100–200 µm granulometric fraction was used for ESR measurements. For powder samples weighing less than 500 mg, the 0–100 and 100–200 µm granulometric fractions were combined.

*2.2. ESR analyses* 

The enamel fraction of each sample was split into six to ten aliquots depending on the amount of available starting material. One of them was kept intact (to measure the natural ESR signal) whereas the remaining five to nine aliquots were irradiated with increasing doses 211 (40, 60, 100, 160, 250, 400, 630, 1000, 1500 Gy) using a <sup>60</sup>Co source (LABRA, CEA, Saclay, France).

The ESR analyses were performed at the Muséum national d'Histoire naturelle (MNHN, Paris, France) with an EMX Bruker ESR spectrometer working at room temperature (19 °C) with the following parameters: 5 mW microwave power, 0.1 mT modulation amplitude, 12 mT scan range, 2 min scan time and 100 kHz modulation frequency. The ESR measurements were repeated 4 times for each aliquot. The equivalent doses were calculated 218 from the enamel T1-B2 signal at  $g = 2.0018$  (Grün, 2000) using OriginPro 8 software (OriginLab Corporation, Northampton, USA). A mean value of ESR intensity and associated standard deviation are obtained from the four measurements and plotted as a function of the irradiation dose for the construction of the dose response curve (DRC).

The DRCs were obtained using a single saturation exponential (SSE) function (Yokoyama et al., 1985) and weighted by the inverse of the squared ESR intensity (1/I²).

#### *2.3. Dose rate evaluation and age calculation*

226 The dental tissues are sampled in order to measure the  $^{238}$ U content and isotopic ratios  $(^{234}U/^{238}U$  and  $^{230}Th/^{234}U$ ) required for U uptake modelling and dose rate reconstruction. U-uptake history can be reconstructed by the calculation of the U-uptake parameter, the '*p*-value' (Grün et al., 1988), ranging from −1 (corresponding to an early uptake, EU) to positive values (corresponding to a recent uptake, RU). For the U-series model (US; Grün et al., 231 1988), <sup>234</sup>U/<sup>238</sup>U and <sup>230</sup>Th/<sup>234</sup>U values are mandatory for determining the *p*-value, while the EU model does not require the computation of isotopic ratios for U-uptake modeling because the *p*-value is fixed to −1. The EU model assumes that the system remained closed during burial after the initial uptake of U and thus provides a minimum age based on the U-content measured in the dental tissues. On the other hand, the RU model, when assuming a dose rate equals to 0, provides a maximum age. Therefore, analyses are done on the enamel and on the dentine and, if present, on the cement attached to the enamel. If the tooth is covered by cement (e.g., *Equus*), the beta dose rate is calculated from the U-content measured in the cement. For *Capra ibex* and *Rhinoceros* teeth, the external side of the enamel was in contact 240 with the sediment so the beta dose rate is calculated from <sup>238</sup>U, <sup>232</sup>Th and <sup>40</sup>K content measured in the sediment.

U-Th analyses Dental tissues were analyzed using either inductively coupled plasma-mass 243 spectrometry (ICP-MS;  $n = 30$ ) or alpha spectrometry ( $n = 5$ ). U and Th isotopes were extracted and purified from each dental tissue following the protocol described in Richard et al. (2015). Measurements were performed using a Multi-Collector ICP-MS Nu instrument (Geotop, UQAM, Montreal, Canada) following Ponte et al. (2017), or a Quadripole ICP-MS Thermo Fisher Scientific Xseries II (LSCE, Gif-sur-Yvette, France) following Douville et al. (2010). Alpha spectrometry measurements were performed at MNHN (Paris, France) following the procedure described in Bischoff et al. (1988).

Sediment beta dose rate determination For equid teeth, the beta dose rate was calculated considering the dentine-enamel-cement configuration, whereas for *Capra ibex* (GH 18) and *Rhinoceros* (GH 21) teeth, the beta dose rate was calculated using a dentine-enamel-sediment configuration (Table 1). Sediment samples (~100 g) from these units were measured at the 254 MNHN with a high-resolution, low-background Ge detector, to determine their  $^{238}$ U,  $^{232}$ Th, 255 and K contents. Beta dose rates were derived from these values taking into account the conversion factors of Adamiec and Aitken (1998) available in the DATA program (Grün, 2009).

258 In situ gamma dosimetry The gamma dose rate was measured in situ using  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:C dosimeters. They were inserted in the section to record the environmental dose rate as a function of water content fluctuation for approximately a year (Fig. 5). A travel dosimeter was used to estimate the dose recorded between the time of removal and the measurement in the 262 laboratory by these dosimeters.

Cosmic dose rate The cosmic dose rate in Geißenklösterle was difficult to evaluate, for two reasons: the roof thickness varies from 4 to 15 m and the large entrance of the cave, opening to the west, overlooks the excavation area (Figs. 5 and 6). Most of the samples were thus shielded to the east, but received a cosmic contribution from the western part of the cave. It is thus necessary to estimate the cosmic dose rate for each tooth sample and dosimeter according its x, y and z coordinates. The cosmic dose was then modelled in 3D taking into account the geometry of the cave and the varying thickness of deposits and limestone present above the sample. The exposure of each sample to the cosmic rays can be modelled considering that the 271 angular repartition of the cosmic dose rate follows the  $\cos^2\theta$  of the angle measured in relation to the vertical axis. The contribution area of the cosmic rays can be represented as a half sphere, divided into four cardinal directions (north, south, east, west). The roof thickness was 274 measured according three angles of incidence:  $0^{\circ}$ -30°,  $30^{\circ}$ -60° and  $60^{\circ}$ -90° in the four 275 cardinal directions (Fig. 6). The cone from  $0^{\circ}$  to  $30^{\circ}$  provides around 65% of the cosmic dose 276 rate, while cones with angles between  $30^{\circ}$  and  $60^{\circ}$  and  $60^{\circ}$  and  $90^{\circ}$  provide around  $30\%$  and 277 5% of the cosmic dose rate, respectively. The cosmic dose rate was calculated according to 278 Prescott and Hutton (1988).

279 ESR age calculation Ages were calculated with  $1 \sigma$  error range using the DATA program 280 (Grün, 2009). The following parameters were used for computation: an alpha efficiency of 281 0.13 ± 0.02 (Grün and Katzenberger-Apel, 1994) was considered; Monte-Carlo beta 282 attenuation factors (Brennan et al., 1997) used; a water content (weight %) of 0% for the 283 enamel and of  $7 \pm 5\%$  for the dentine and cement assumed. The water content (wet weight  $\%$ ) 284 in the sediment was determined by weighing the sample before and after drying in an oven for 285 one week at 40 °C. The following values were used for individual age calculations:  $15 \pm 5\%$ 286 for GHs 15 and 16;  $13 \pm 5\%$  for GH 17;  $10 \pm 5\%$  for GHs 18 and 21 (7–15% measured).

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#### 288 **3. Results**

289 U-series analyses were conducted in order to reconstruct the U-uptake history of the 290 dental tissues that might affect the dose rate (Table 2). U-content is low for all samples, 291 ranging from 0.0023 to 0.3650 ppm. For samples with extremely low U and Th contents,  $^{234}$ U 292 and <sup>230</sup>Th signals were too weak and isotopic ratios  $(^{234}U/^{238}U, ^{230}Th/^{234}U$  and <sup>230</sup>Th/<sup>232</sup>Th) 293 were not determined. Otherwise,  $^{234}U^{238}U$  activity ratios were superior to 1 and  $^{230}Th^{234}U$ 294 inferior to 1, as expected. In Geißenklösterle cave, the low U-content of the teeth generates 295 dose rates close to 1  $\mu$ Gy·a<sup>-1</sup> for most of the dental tissues. When isotopic ratios were 296 determined, US-ESR ages were calculated but, as expected, were not distinguishable from the 297 EU-ESR ages, except for sample GK 57-2609 (SOM Table S1). More specifically, this tooth 298 has the highest U-content measured in the enamel  $(0.2583 \pm 0.0016$  ppm). Its internal  $(\alpha + \beta)$ 299 dose rate is consequently not negligible and requires the use of the US model. Considering these observations, the ages can be calculated using the EU model, except for sample GK 57- 2609, which requires a modelling of the internal dose rate using the US model.

 $2^{238}$ U,  $2^{32}$ Th and  $40$ K contents measured in the sediment and used for the calculation of the beta dose rate received by the teeth sampled in GHs 18 and 21 are given in Table 3. The radioelements content is lower in GH 21 than in GH 18, while the gamma dose rates recorded 305 by the dosimeters (Table 4) are similar (60 and 67  $\mu$ Gy/a, respectively). Nonetheless, we can observe that both gamma dose rate and radioelements content measured in the sediment from GHs 15, 17, 18 and 21 tend to decrease with depth (Tables 3 and 4). The mechanism that produced these results needs further investigation. No disequilibrium was observed except for the sediment from GH 15 for which a slight disequilibrium (lower than 10%) was determined.

The values recorded by the dosimeters correspond to a low radioactive environment, 311 with gamma dose rates ranging from 60  $\mu$ Gy·a<sup>-1</sup> (GH 21) to 145  $\mu$ Gy·a<sup>-1</sup> (GH 15; Table 4). Three dosimeters were placed each in GHs 15 and 17, and for each GH a mean value with its standard deviation was calculated. However, we noticed that the gamma dose values recorded 314 in GH 15 were scattered, from 66 to 145  $\mu$ Gy·a<sup>-1</sup>. Since the sedimentary matrix containing the teeth has been excavated, the gamma dose rate is measured from the remaining sections. It is thus not possible to determine if the teeth belonged to the lower or the higher dosimetric environment. The mean value of  $108 \pm 42 \mu Gy \cdot a^{-1}$  was thus used for the age calculation, but could be unrepresentative of the dose rate underwent by the samples unearthed from this GH (see discussion on the spatial variation of the gamma dose rate in Richter, 2007). For GH 16, 320 which was entirely excavated, a mean value of  $106 \pm 23 \text{ uGv} \cdot a^{-1}$  was computed from the values recorded in GHs 15 and 17. For GHs 18 and 21, only one dosimeter could be inserted in each unit, due to the collapse of the section during fieldwork.

323 Ages, equivalent doses and dose rates are presented in Table 5. They range from  $94 \pm 1$ 324 10 ka (base of the sequence, GH 21) to  $35 \pm 8$  ka (top of the sequence, GH 15). Dose response

curves are available in SOM Figs. S2–S14. GK 57-2609 is the only sample for which the EU-326 ESR (56  $\pm$  6 ka) and the US-ESR (73  $\pm$  9 ka) ages are significantly different. In this specific case, the EU-ESR age represents the minimum age, and the US-ESR age is thought to be more reliable. Indeed, if we consider the U-contents measured in all the enamels, this one is 329 the highest ( $\approx$ 0.26 ppm), inducing a significant internal ( $\alpha + \beta$ ) dose rate. *P*-values for this 330 sample derived from the US model are  $0.80 \pm 0.40$  for the enamel and  $2.15 \pm 0.17$  for the dentine, describing sublinear and recent uptakes, respectively. In contrast, for all other samples, EU, LU (linear uptake, Ikeya, 1982), RU and US-ESR ages are not distinguishable, with differences lower than 6% (SOM Table S1).

334 Table 5 shows that equivalent doses  $(D_e)$  obtained using a SSE function (Fig. 7) are 335 low, ranging from 5 to 12 Gy. To avoid equivalent dose overestimation, Duval and Grün 336 (2016) recommended using a maximum irradiation dose  $(D_{max})$  up to 10 times the  $D_e$ . Since 337 D<sub>max</sub> is 1500 Gy, D<sub>max</sub>/D<sub>e</sub> values are high, included between 131 and 302 (SOM Table S2). To 338 test the impact of high irradiation steps on the  $D<sub>e</sub>$  estimation, equivalent doses were calculated 339 by removing the highest experimental points on the dose response curve for three samples 340 whose  $D_e$  are inferior to 11 Gy. The maximum difference between the  $D_e$  is less than 3% 341 (SOM Table S3), suggesting that high  $D_{max}/D_e$  values do not impact the determination of the 342 De for the samples analyzed in this study. Moreover, for GH 18, no significant variation can 343 be observed between  $D_e$  obtained in this study, ranging from 8.7  $\pm$  0.2 to 10.9  $\pm$  0.4, with 344 those published by Richter et al. (2000), ranging from  $8.1 \pm 0.7$  to  $10 \pm 0.6$  Gy and obtained 345 with exponential dose steps from 5 to 960 Gy. These low De values are correlated with low U-346 content and environmental dose rates. Indeed, gamma (plus beta, if applicable) dose rates From the sediment are very low, ranging from  $64 \pm 15$  (GH 21) to  $108 \pm 42 \mu Gy \cdot a^{-1}$  (GH 15). However, the cosmic dose rate is significant, varying from 38 to 99  $\mu$ Gy·a<sup>-1</sup> depending on 349 sample location. It represents up to 47% (GK 75-426) of the dose rate. By contrast, the sum of the dose rates from each tissue does not exceed 6%, at the exception of GK 57-2609, which rises to 13% (Fig. 8).

#### **4. Discussion**

These ESR ages obtained for the lower part of the stratigraphic sequence provide new information regarding the Middle Paleolithic occupations of the cave (Fig. 9). The two ESR 356 results obtained for GH 21 (AH VII), of  $94 \pm 10$  ka (GK 47-583) and 73  $\pm$  9 ka (GK 57-357 2609), indicate an occupation during the MIS 5, and a weighted mean age of  $82 \pm 9$  ka may be calculated. However, these two ages do not overlap. Two hypotheses could explain the age difference: 1) the deposition time of this layer is in the order of 10–40 ka or 2) diagenetic processes during burial could have altered GK 57-2609, which has a higher U-content (around 0.26 ppm) in the enamel than in the dentine. Indeed, enamel is less porous than dentine and cement and so the U-content is generally lower (e.g., Eggins et al., 2003; Kohn, 2008). In the case of GK 57-2609, the U-content of 0.26 ppm in the enamel is not well understood, especially if we consider the other dated samples, for which the U-content does not exceed 0.03 ppm. However, this hypothesis requires further investigation to assess the preservation state of the tooth sample. Nonetheless, if we take into account the RU-ESR age of this sample 367 (maximum age calculated assuming a dose rate from the dental tissues equal to 0), of  $83 \pm 9$ ka, it is in agreement with both EU-ESR and RU-ESR ages for GK 47-583 (SOM Table S1), 369 of  $94 \pm 10$  ka (EU) and  $100 \pm 12$  ka (RU).

370 For GH 18 (AH IV), the new ESR ages, ranging from  $65 \pm 8$  ka to  $55 \pm 6$  ka, are 371 somewhat older than previous ESR ages (Richter et al., 2000; from  $53 \pm 5$  ka to  $36 \pm 5$  ka; 372 Fig. 10). The weighted mean age is  $60 \pm 4$  ka, while the one published by Richter et al. (2000) 373 is  $43 \pm 4$  ka. The equivalent doses reported in Richter et al. (2000) for the dated teeth range 374 from 8.1  $\pm$  0.7 to 10  $\pm$  0.6 Gy and are similar to those obtained in the present study, ranging 375 from  $8.7 \pm 0.2$  to  $10.9 \pm 0.4$  (Table 5). The beta dose rate from the sediment was calculated from delayed neutron counting (U) and neutron activation (Th, K; Richter et al., 2000) and 377 ranged from 79 to 151  $\mu$ Gy·a<sup>-1</sup>. However, our beta dose rate (measured using laboratory 378 gamma ray spectrometry) is lower, ranging from  $32 \pm 4$  to  $44 \pm 6 \mu Gy \cdot a^{-1}$ . While in gamma spectrometry bulk samples, which include small pebbles, rocks, bones, etc. are analyzed, the small sample for NAA consisted of clayey sediment only. This was taken as a more realistic scenario by Richter et al. (2000) considering the short ranged beta contribution (around 2 mm) and that teeth are usually encased by clayey sediments. It could explain why the beta dose rates measured by Richter et al. (2000) are much higher than those we measured using laboratory gamma ray spectrometry. Moreover, cave deposits are generally heterogeneous dosimetric environments, due to the presence of limestone rocks for instance. Lateral variation of the gamma dose rate should be also considered as a potential source for the age 387 discrepancy. Indeed, a value of 67  $\mu$ Gy·a<sup>-1</sup> was measured in GH 18 for the present study, while previous published ages by Richter et al. (2000) were calculated using a value of 39  $\mu$ Gy·a<sup>-1</sup>. These results suggest that Neanderthals inhabited the cave not only at the beginning of MIS 3 but during MIS 4 as well. These ages, obtained for the lower part of the sequence, are systematically older than  ${}^{14}C$  ages. Their antiquity, combined with the fact that  ${}^{14}C$  ages obtained previously do not increase as a function of depth, indicate an occupation time beyond the limit of radiocarbon, i.e., older than 50 ka (Fig. 10). De facto, uncalibrated  $^{14}C$  age 394 published by Higham et al. (2012) for GH 21, of  $48.6 \pm 3.2$  ka, should be considered as a minimum age. The two sets of results obtained for GHs 21 and 18 allow, for the first time, to constrain Neanderthal occupation at Geißenklösterle Cave and demonstrate the antiquity of the settlement in the cave, starting during MIS 5 (Fig. 9).

398 Ages derived for the transitional horizon (GH 17, AH IIIc) range from  $48 \pm 6$  ka to  $44$  $\pm$  5 ka, and are in agreement with <sup>14</sup>C ages (Fig. 10), except for the age published by Conard and Bolus (2003). This date was not obtained using the ultrafiltration protocol, used to remove modern contaminants, which could explain why it appears younger than those published by Conard and Bolus (2008) and Higham et al. (2012). The fact that this unit is almost sterile suggests an interruption of human activities in the cave, probably during a cold 404 phase. Indeed, the weighted mean age of this unit  $(46 \pm 3 \text{ ka})$  coincides with Greenland Stadial (GS) 12 or 13. Stratigraphic discontinuities between Middle and Upper Paleolithic layers have also been identified elsewhere in Europe (e.g., Spain: Mallol et al., 2012; Italy: Peresani, 2008). This supports the hypothesis of a regional extinction or substantial depopulation of Neanderthals in the Swabian Jura prior to the arrival of modern humans (Conard et al. 2006). Radiocarbon ages indicate that early Aurignacian deposits predate Heinrich Stadial 4 (around 40–38 ka; Higham et al., 2012). Taking into account all available chronological data, GH 17 could have been deposited during Heinrich Stadial 5, which took place between ca. 49–47 ka (Svensson et al., 2008).

For the Aurignacian layers (GHs 15 and 16, AHs III, IIIa and IIIb), ESR ages are in 414 agreement with <sup>14</sup>C and TL data (Fig. 10). Three ages are available for GH 15, of 40  $\pm$  10 ka,  $38 \pm 8$  ka and  $35 \pm 8$  ka but have a large error due to the large variation of the gamma dose 416 rate recorded by the dosimeters (see Table 4). A weighted mean of  $37 \pm 5$  ka may be obtained. 417 These ages are constrained by the two other ages obtained for GH 16 (50  $\pm$  8 ka and 34  $\pm$  4; 418 Fig. 9), for which a weighted mean age of  $37 \pm 8$  ka may be calculated. The difference between these two ages could be explained by their stratigraphic position or by the spatial variation of the in situ gamma dose rate, which cannot be directly measured in GH 16. The youngest is attributed to a tooth sampled at a depth of 3.43 m, whereas the oldest age is associated with a sample found at a depth of 3.70 m (Fig. 9). However, since this layer was entirely excavated, the gamma dose rate cannot be measured in situ and no sediment was available for laboratory gamma ray spectrometry. It was thus not possible to assess the degree of heterogeneity for GH 16. Considering that the ages for this layer were computed using the gamma dose rate recorded in both GHs 15 and 17, we do not exclude a local variation of the gamma dose rate as observed in GH15, which would account for the age discrepancy. 428 Considering ESR ages obtained for both GHs 15 and 16, a weighted mean age of  $37 \pm 3$  ka may be obtained for the lower Aurignacian layers.

#### **6. Conclusions**

ESR dating was used to date herbivorous teeth from Middle Paleolithic and early Aurignacian horizons in order to extend the chronological framework of the Geißenklösterle 434 sequence (Swabian Jura, Germany). The ESR results were compared with previous  ${}^{14}C$ , ESR and TL results available for the Aurignacian and the uppermost Middle Paleolithic layers. They demonstrate the potential and limitations of such application in a heterogeneous dosimetric environment. Indeed, the difficulty in reconstructing the gamma dose rate and estimating the cosmic contribution to the annual dose induce large uncertainties in the ages 439 obtained for the early Aurignacian deposits. The agreement between TL and/or  ${}^{14}C$  and ESR ages obtained on GH 17 and early Aurignacian deposits bolsters the hypothesis of an early occupation by *H. sapiens* before 40 ka, and supports ESR ages obtained from Middle Paleolithic layers, which gives an age estimate for the onset of occupation in the cave, starting from MIS 5.

From a methodological perspective, the low U-content in most of the dental tissues allows to calculate EU-ESR ages without encountering the usual uncertainties resulting from the reconstruction of the unknown U-uptake history. However, considering that the dose rates in dental tissues are negligible and that the gamma doses are also low, the cosmic dose rate represents up to 47% of the annual dose. Therefore, it was computed specifically for each sample considering the morphology of the cave and the distance between each sample and the

entrance. This study highlighted two aspects of the application of ESR dating to Late Pleistocene karstic sites: 1) a large dependence on the gamma and cosmic dose rates for which variations may significantly affect the ages; and 2) the low uranium concentrations induce a negligible internal dose rate, thus allowing the calculation of meaningful and finite EU-ESR age results that are indistinguishable from the US-ESR estimates.

In the context of the end of the Middle Paleolithic, the study of Neanderthal occupations within a chronological framework at Geißenklösterle cave is fundamental to assess the spatiotemporal disappearance of this population from Europe, as well as to understand group dynamics during the Late Pleistocene. ESR ages obtained in this study suggest that groups of Neanderthals occasionally occupied the cave between MIS 5 and 4, 460 from 94  $\pm$  10 ka to 55  $\pm$  6 ka, and provide for the first time chronological constraints for the base of the sequence.

462 Moreover, the presence of an almost sterile layer (GH 17), dated by ESR to  $46 \pm 3$  ka (weighted mean age), could represent the end of human activities and the abandonment of the 464 site. Taking into account that the  ${}^{14}C$  ages for early Aurignacian deposits are older than Heinrich Stadial 4 by several thousands of years, we can conclude that *H. neanderthalensis* likely occupied the site until Heinrich Stadial 5, before the arrival of *H. sapiens* in the area. This raises questions about the relationship between these two species in this region. Did they really meet in the Swabian Jura? This is a core question, especially when we focus on the absence of transitional industries in this area, and the few human remains documented in the region. A complete chronological study of the Middle Palaeolithic sites of Swabian Jura needs to be carried out in the future, especially for major cave sites such as Hohle Fels, Sirgenstein, Kogelstein and Große Grotte to establish a well-defined chronological framework for the Swabian Middle Paleolithic.

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1 : Kogelstein 2 : Hohle Fels 3 : Sirgenstein 4 : Geißenklösterle 5 : Brillenhöhle 6 : Große Grotte 7 : Haldenstein 8 : Bockenstein (Bockstein-Höhle, Bocksteinloch, Bocksteinschmiede and Bockstein-Törle) 9 : Hohlenstein (Stadel and Bärenhöhle) 10: Vogelherd 11 : Heidenschmiede

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# Sample





## **Table 1**

Sample type, laboratory number, geological (GH) and archaeological (AH) horizons, coordinates and taxonomic determination of samples from Geißenklösterle. The map and grid of the cave are available in Figure 5.

Type	Lab $N^{\circ}$	GH	AH	Square	X(m)	Y(m)	Z(m)	Taxon	Grain size $(\mu m)^a$
Tooth	GK 75-426	15	Ш	75	25.57	29.13	$-3.52$	<i>Equus</i> sp.	$100 - 200$
Tooth	GK 78-1278	15	<b>IIIb</b>	78	28.49	29.85	$-3.48$	<i>Equus</i> sp.	$100 - 200$
Tooth	GK 76-1080	15	Шa	76	26.59	29.72	$-3.55$	<i>Equus</i> sp.	$0 - 200$
Sediment	GK.sed.GH15	15	Ш	99	29.21	31.12	$-3.30$		
Tooth	GK 47-380	16	Шa	47	27.41	26.66	$-3.70$	<i>Equus</i> sp.	$100 - 200$
Tooth	GK 69-976	16	IIIb	69	29.83	28.72	$-3.43$	<i>Equus</i> sp.	$100 - 200$
Tooth	GK 58-909	17	<b>III</b> c	58	28.19	27.85	$-3.70$	<i>Equus</i> sp.	$100 - 200$
Tooth	GK 47-397	17	<b>III</b> c	47	27.42	26.57	$-3.75$	<i>Equus</i> sp.	$100 - 200$
Tooth	GK 77-1607	17	<b>III</b> c	77	27.05	29.06	$-3.85$	<i>Equus</i> sp.	100200
Sediment	GK.sed.GH17	17	<b>III</b> c	69	29.17	28.37	$-3.82$		
Tooth	GK 47-447	18	IV	47	27.06	26.96	$-4.12$	Capra ibex	$0 - 200$
Tooth	GK 47-451	18	IV	47	27.44	26.43	$-4.05$	Capra ibex	$100 - 200$
Tooth	GK 78-1511	18	IV	78	28.50	29.05	$-4.09$	Capra ibex	$0 - 200$
Sediment	GK.sed.GH18	18	IV	69	29.16	28.28	$-4.13$		
Tooth	GK 57-2609	21	VII	57	27.13	27.96	$-4.39$	<i>Equus</i> sp.	$0 - 200$
Tooth	GK 47-583	21	VII	47	27.83	26.20	-4.42	<i>Rhinoceros</i> sp.	$100 - 200$
Sediment	GK.sed.GH21	21	VII	69	29.19	28.63	-4.57		

a ESR analyses are commonly carried out on the 100–200 µm granulometric fraction and U-Th on the 0–100 µm fraction. For powder samples weighing less than 500 mg, the 0–100 and 100-200 µm granulometric fractions were combined and ESR measurements were performed before U-Th analysis.

#### **Table 2**



U-Th data (U-content and isotopic ratios) and initial and removed thickness on side 1 (dentine) and side 2 (cement or sediment).

D  $0.0664 \pm 0.0005$   $1.3057 \pm 0.0185$   $0.2460 \pm 0.0123$ 

Abbreviations:  $nd = not detectable$ ;  $E = channel$ ;  $D = dentine$ ;  $C = cement$ ;  $GH = geological horizon$ .

<sup>a</sup> These samples were analysed using alpha spectrometry according to a chemical procedure detailed in Bischoff et al. (1988).

## **Table 3**

sediment sample is available in Figure 5.										
Sample	GH	$^{238}$ U (ppm)	$232$ Th (ppm)	$^{40}$ K (%)						
GK.sed.GH15	15	$0.94 \pm 0.07$	$2.89 \pm 0.10$	$0.30 \pm 0.01$						
GK.sed.GH17	17	$0.54 \pm 0.07$	$2.37 \pm 0.01$	$0.22 \pm 0.01$						
GK.sed.GH18	18	$0.58 \pm 0.07$	$1.59 \pm 0.09$	$0.16 \pm 0.01$						
GK.sed.GH21	21	$0.20 \pm 0.05$	$0.81 \pm 0.06$	$0.06 \pm 0.01$						

 $238$ U,  $232$ Th and  $40$ K measured using a HPGe gamma spectrometer. The position of each

Abbreviation: GH = geological horizon.

### **Table 4**

In situ gamma dosimetry. Dosimeter number and associated section, coordinates and dose rate (see map of the cave and photographs of the section in Fig. 5).



Abbreviation:  $GH =$  geological horizon.

<sup>a</sup>This value corresponds to a mean value obtained from measurements in both GHs 15 and 17 (see text).

<sup>b</sup> A 20% error range was estimated for these values, from gamma dose variations observed in GHs 15 and 17.

# **Table 5** Equivalent doses, dose rates and EU-ESR or US-ESR (bold) ages.



Abbreviation: GH = geological horizon.

<sup>a</sup> A 10% error was estimated for the cosmic dose.

<sup>b</sup> US-ESR age with associated *p*-values of  $0.80 \pm 0.40$  (enamel) and  $2.15 \pm 0.17$  (dentine).