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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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**Keywords:** ESR dating; Middle Paleolithic; early Aurignacian; Neanderthal; *Homo sapiens*; Swabian Jura

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28 Swabian Jura

29

30 **Abstract**

31 Geißenklösterle Cave (Germany) is one of the most important Paleolithic sites in  
32 Europe, as it is characterized by human occupation during the Middle and Early Upper  
33 Paleolithic. Aurignacian layers prior to 37–38 ka cal BP feature both musical and figurative  
34 art objects that are linked to the early arrival in Europe of *Homo sapiens*. Middle Paleolithic  
35 layers yielded lithic artifacts attributed to *Homo neanderthalensis*. Since human occupation at  
36 the site is attributed to both Neanderthals and modern humans, chronology is essential to  
37 clarify the issues of Neanderthal disappearance, modern humans expansion in Europe, and the  
38 origin of the Aurignacian in Western Europe. Electron spin resonance (ESR) dating was  
39 performed on fossil tooth enamel collected from the Middle Paleolithic layers, which are  
40 beyond the radiocarbon dating range, and from the nearly sterile ‘transitional’ geological  
41 horizon (GH) 17 and the lower Aurignacian deposits, to cross-check ESR ages with previous  
42 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  
45 agreement with previous dating results, thus supporting the reliability of ESR chronology for  
46 the base of the sequence where dating comparisons are not possible. These results suggest that  
47 Neanderthals occupied the site from Marine Isotope Stage (MIS) 5 to the second half of MIS  
48 3 and confirm the antiquity of early Aurignacian deposits. The presence of an almost sterile  
49 layer that separates Middle and Upper Paleolithic occupations could be related to the  
50 abandonment of the site by Neanderthals, probably during Heinrich Stadial 5 (ca. 49–47 ka),

51 thus before the arrival of *H. sapiens* in the area around 42 ka cal BP. These dates for the  
52 Middle Paleolithic of the Swabian Jura represent an important contribution to the prehistory  
53 of the region, where nearly all of the excavations were conducted decades ago and prior to the  
54 development of reliable radiometric dating beyond the range of radiocarbon.

55

## 56 **1. Introduction**

### 57 *1.1. Dating the end of the Middle Paleolithic and the beginning of the Upper Paleolithic*

58 The Middle to Upper Paleolithic transition has been the subject of numerous studies  
59 and has been a topic of lively debate for the past thirty years (e.g., White, 1982; Mellars,  
60 1999; Conard, 2006; Villa and Roebroeks, 2014). Different hypotheses have been proposed to  
61 explain the disappearance of Neanderthals (*Homo neanderthalensis*) and the arrival and  
62 expansion of anatomically modern humans (*Homo sapiens*) in Eurasia during the Late  
63 Pleistocene (e.g., Mellars, 2004; Hockett and Haws, 2005; Conard et al., 2006; Verpoorte,  
64 2006; Müller et al., 2011; Harvati-Papatheodorou, 2013). The cultural interactions between  
65 these two species are still debated and two main hypotheses are often invoked to explain the  
66 changes observed in the technical behavior during this crucial period. The presence of  
67 ‘transitional industries’ from around 45 ka to 30 ka (e.g., Douka et al., 2014), or as early as 48  
68 ka (Richter et al., 2009) across Europe has been interpreted either as the result of  
69 Neanderthals and modern humans ‘cohabitation’, which led to cultural exchanges (the  
70 acculturation hypothesis; e.g., Hublin et al., 1996; Mellars, 2005) or as an endogenous  
71 development attributed to Neanderthals (the multiple-species model, e.g., d’Errico et al., 1998;  
72 d’Errico, 2003; Zilhão et al., 2006). The emergence of the Aurignacian, which broadly  
73 coincides with the first known *H. sapiens* in Western Europe (Higham et al., 2011, 2012) also  
74 raises questions about the development of both figurative and musical art out of Africa (e.g.,  
75 Conard, 2010, 2015). During the last twenty years, the advances in dating methods have been

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

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

101 using radiocarbon (Conard and Bolus, 2003, 2008; Higham et al., 2012), which confirms  
102 thermoluminescence (TL) ages (Richter et al., 2000).

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

109

## 110 *1.2. Geißenklösterle Cave*

111 Geißenklösterle Cave is located near Blaubeuren in the Swabian Jura (Baden-  
112 Württemberg, Germany), a region of low mountains in southwest Germany (Fig. 1). The cave  
113 entrance faces the Ach Valley and opens above the river Ach, a Danube tributary. The region  
114 is famous for some of the most ancient examples of both musical and figurative art, attributed  
115 to *H. sapiens* (e.g., Hahn, 1971, 1988; Conard, 2003, 2009; Conard et al., 2009; Kind et al.,  
116 2014). Middle Paleolithic occupations, attributed to Neanderthals (Conard et al., 2012), are  
117 also numerous in the Swabian Jura. At Geißenklösterle Cave, it has been suggested that these  
118 two human species successively occupied the cave, making this site and its chronology  
119 particularly important for the debate on the origins of the Aurignacian in Western Europe  
120 (Sauvet et al., 2008; Hublin, 2012; Floss, 2017).

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

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

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

143         The faunal remains documented in the Middle Paleolithic and early Aurignacian layers  
144 are associated to a cold climate, related to a ‘mammoth steppe environment’ (Guthrie, 1990),  
145 as attested by the presence of reindeer (*Rangifer tarandus*), mammoth (*Mammuthus*  
146 *primigenius*), woolly rhino (*Coelodonta antiquitatis*) and wild horse (*Equus ferus*). Given the  
147 abundant presence of cave bear (*Ursus spelaeus*), it seems that Geißenklösterle Cave was  
148 used as a hibernation den (Münzel and Conard, 2004). The high concentration in mammoth  
149 remains is likely linked to the use of ivory by humans during the Aurignacian period, as  
150 documented by the presence of ivory points (Münzel, 2001). The density of faunal remains is



151 much lower in the Middle Paleolithic layers, perhaps in relation with the size of the human  
152 groups that occupied the cave. The population density and the occupation intensity would  
153 have been lower during the Middle Paleolithic than during the Aurignacian period (Münzel  
154 and Conard, 2004; Conard et al., 2012).

155         The exceptional archaeological sequence of Geißenklösterle was the subject of several  
156 dating studies, mainly focused on the Upper Paleolithic layers, which are of great importance  
157 with regard to the emergence and development of the Aurignacian culture. Hypotheses such  
158 as the ‘Kulturpumpe’ and the ‘Danube corridor’ (according to which the Danube Valley  
159 would have been the main migration route between Southeastern Europe and the Swabian  
160 Jura; Conard and Bolus, 2003) are based on the chronology established for the lower  
161 Aurignacian deposits at Geißenklösterle. Chronological data thus played a key role in the  
162 interpretation of human dynamics during the Late Pleistocene and radiocarbon dating was  
163 consequently widely applied to samples from Upper Palaeolithic layers. Richter et al. (2000)  
164 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,  
166 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 =  $43.3 \pm 4.0$   
168 ka). They suggest that the last Neanderthals occupied the site between the end of MIS 4 and  
169 the beginning of MIS 3.

170         A large number of  $^{14}\text{C}$  ages were obtained on bones from several laboratories, mostly  
171 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  $^{14}\text{C}$  ages have  
173 been reported from the Middle Paleolithic levels, ranging from around 45 (AH VIII) to 37 ka  
174 cal BP (AH IV). It has been argued by Conard (2002) and Conard and Bolus (2003) that  
175 important variations in atmospheric  $^{14}\text{C}$  concentration between 50 ka and 30 ka were the

176 cause for some of the younger radiocarbon ages, based on the data obtained in North Atlantic  
177 planktonic foraminifera (Voelker et al., 2000), Japanese varves (Kitagawa and van der Plicht,  
178 1998) and stalagmites from the Bahamas (Beck et al., 2001). Called the ‘Middle Paleolithic  
179 Dating Anomaly’, this phenomenon supposedly was responsible for interpretations of Middle  
180 and Upper Paleolithic occupations overlapping in time, and thus exaggerating the  
181 ‘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 <sup>14</sup>C  
183 determinations of ultrafiltered collagen. Their results confirmed the antiquity of the  
184 Aurignacian layers, as suggested by Richter et al. (2000), which were deposited as early as 40  
185 ka cal BP. More specifically, Higham et al. (2012) used Bayesian modelling to date the start  
186 of the early Aurignacian between 43 and 42 ka cal BP. However, regarding the transitional  
187 horizon (GH 17) and the Middle Paleolithic layers, only a few ages have been reported.  
188 Taking into account available data for the Middle to Upper Paleolithic levels, the results  
189 indicate that the early Aurignacian deposits and the almost sterile GH 17 are older than 40 ka  
190 cal BP, and the Middle Paleolithic layers range between around 52 ka cal BP (AH VII) and 35  
191 ka cal BP (AH IV; Conard and Bolus, 2003, 2008; Higham et al., 2012).

192

## 193 **2. Materials and methods**

### 194 *2.1. Sampling and sample preparation*

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

200 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  $\mu\text{m}$  was removed from each  
202 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  $\mu\text{m}$  granulometric  
204 fraction was used for ESR measurements. For powder samples weighing less than 500 mg, the  
205 0–100 and 100–200  $\mu\text{m}$  granulometric fractions were combined.

206

## 207 2.2. ESR analyses

208 The enamel fraction of each sample was split into six to ten aliquots depending on the  
209 amount of available starting material. One of them was kept intact (to measure the natural  
210 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  $^{60}\text{Co}$  source (LABRA, CEA, Saclay,  
212 France).

213 The ESR analyses were performed at the Muséum national d'Histoire naturelle  
214 (MNHN, Paris, France) with an EMX Bruker ESR spectrometer working at room temperature  
215 (19 °C) with the following parameters: 5 mW microwave power, 0.1 mT modulation  
216 amplitude, 12 mT scan range, 2 min scan time and 100 kHz modulation frequency. The ESR  
217 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  
219 (OriginLab Corporation, Northampton, USA). A mean value of ESR intensity and associated  
220 standard deviation are obtained from the four measurements and plotted as a function of the  
221 irradiation dose for the construction of the dose response curve (DRC).

222 The DRCs were obtained using a single saturation exponential (SSE) function  
223 (Yokoyama et al., 1985) and weighted by the inverse of the squared ESR intensity ( $1/I^2$ ).

224

### 225 2.3. Dose rate evaluation and age calculation

226 The dental tissues are sampled in order to measure the  $^{238}\text{U}$  content and isotopic ratios  
227 ( $^{234}\text{U}/^{238}\text{U}$  and  $^{230}\text{Th}/^{234}\text{U}$ ) required for U uptake modelling and dose rate reconstruction. U-  
228 uptake history can be reconstructed by the calculation of the U-uptake parameter, the ‘ $p$ -  
229 value’ (Grün et al., 1988), ranging from  $-1$  (corresponding to an early uptake, EU) to positive  
230 values (corresponding to a recent uptake, RU). For the U-series model (US; Grün et al.,  
231 1988),  $^{234}\text{U}/^{238}\text{U}$  and  $^{230}\text{Th}/^{234}\text{U}$  values are mandatory for determining the  $p$ -value, while the  
232 EU model does not require the computation of isotopic ratios for U-uptake modeling because  
233 the  $p$ -value is fixed to  $-1$ . The EU model assumes that the system remained closed during  
234 burial after the initial uptake of U and thus provides a minimum age based on the U-content  
235 measured in the dental tissues. On the other hand, the RU model, when assuming a dose rate  
236 equals to 0, provides a maximum age. Therefore, analyses are done on the enamel and on the  
237 dentine and, if present, on the cement attached to the enamel. If the tooth is covered by  
238 cement (e.g., *Equus*), the beta dose rate is calculated from the U-content measured in the  
239 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  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  content  
241 measured in the sediment.

242 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  
244 extracted and purified from each dental tissue following the protocol described in Richard et  
245 al. (2015). Measurements were performed using a Multi-Collector ICP-MS Nu instrument  
246 (Geotop, UQAM, Montreal, Canada) following Ponte et al. (2017), or a Quadripole ICP-MS  
247 Thermo Fisher Scientific Xseries II (LSCE, Gif-sur-Yvette, France) following Douville et al.  
248 (2010). Alpha spectrometry measurements were performed at MNHN (Paris, France)  
249 following the procedure described in Bischoff et al. (1988).

250 Sediment beta dose rate determination For equid teeth, the beta dose rate was calculated  
251 considering the dentine-enamel-cement configuration, whereas for *Capra ibex* (GH 18) and  
252 *Rhinoceros* (GH 21) teeth, the beta dose rate was calculated using a dentine-enamel-sediment  
253 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  $^{238}\text{U}$ ,  $^{232}\text{Th}$ ,  
255 and  $^{40}\text{K}$  contents. Beta dose rates were derived from these values taking into account the  
256 conversion factors of Adamiec and Aitken (1998) available in the DATA program (Grün,  
257 2009).

258 In situ gamma dosimetry The gamma dose rate was measured in situ using  $\alpha\text{-Al}_2\text{O}_3\text{:C}$   
259 dosimeters. They were inserted in the section to record the environmental dose rate as a  
260 function of water content fluctuation for approximately a year (Fig. 5). A travel dosimeter was  
261 used to estimate the dose recorded between the time of removal and the measurement in the  
262 laboratory by these dosimeters.

263 Cosmic dose rate The cosmic dose rate in Geißenklösterle was difficult to evaluate, for two  
264 reasons: the roof thickness varies from 4 to 15 m and the large entrance of the cave, opening  
265 to the west, overlooks the excavation area (Figs. 5 and 6). Most of the samples were thus  
266 shielded to the east, but received a cosmic contribution from the western part of the cave. It is  
267 thus necessary to estimate the cosmic dose rate for each tooth sample and dosimeter according  
268 its x, y and z coordinates. The cosmic dose was then modelled in 3D taking into account the  
269 geometry of the cave and the varying thickness of deposits and limestone present above the  
270 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  
272 to the vertical axis. The contribution area of the cosmic rays can be represented as a half  
273 sphere, divided into four cardinal directions (north, south, east, west). The roof thickness was  
274 measured according three angles of incidence:  $0^\circ\text{-}30^\circ$ ,  $30^\circ\text{-}60^\circ$  and  $60^\circ\text{-}90^\circ$  in the four

275 cardinal directions (Fig. 6). The cone from 0° to 30° provides around 65% of the cosmic dose  
276 rate, while cones with angles between 30° and 60° and 60° and 90° 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  $0.13 \pm 0.02$  (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).

287

### 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,  $^{234}\text{U}$   
292 and  $^{230}\text{Th}$  signals were too weak and isotopic ratios ( $^{234}\text{U}/^{238}\text{U}$ ,  $^{230}\text{Th}/^{234}\text{U}$  and  $^{230}\text{Th}/^{232}\text{Th}$ )  
293 were not determined. Otherwise,  $^{234}\text{U}/^{238}\text{U}$  activity ratios were superior to 1 and  $^{230}\text{Th}/^{234}\text{U}$   
294 inferior to 1, as expected. In Geißenklösterle cave, the low U-content of the teeth generates  
295 dose rates close to  $1 \mu\text{Gy}\cdot\text{a}^{-1}$  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

300 these observations, the ages can be calculated using the EU model, except for sample GK 57-  
301 2609, which requires a modelling of the internal dose rate using the US model.

302  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  contents measured in the sediment and used for the calculation of  
303 the beta dose rate received by the teeth sampled in GHs 18 and 21 are given in Table 3. The  
304 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  $\mu\text{Gy}/\text{a}$ , respectively). Nonetheless, we can  
306 observe that both gamma dose rate and radioelements content measured in the sediment from  
307 GHs 15, 17, 18 and 21 tend to decrease with depth (Tables 3 and 4). The mechanism that  
308 produced these results needs further investigation. No disequilibrium was observed except for  
309 the sediment from GH 15 for which a slight disequilibrium (lower than 10%) was determined.

310 The values recorded by the dosimeters correspond to a low radioactive environment,  
311 with gamma dose rates ranging from 60  $\mu\text{Gy}\cdot\text{a}^{-1}$  (GH 21) to 145  $\mu\text{Gy}\cdot\text{a}^{-1}$  (GH 15; Table 4).  
312 Three dosimeters were placed each in GHs 15 and 17, and for each GH a mean value with its  
313 standard deviation was calculated. However, we noticed that the gamma dose values recorded  
314 in GH 15 were scattered, from 66 to 145  $\mu\text{Gy}\cdot\text{a}^{-1}$ . Since the sedimentary matrix containing the  
315 teeth has been excavated, the gamma dose rate is measured from the remaining sections. It is  
316 thus not possible to determine if the teeth belonged to the lower or the higher dosimetric  
317 environment. The mean value of  $108 \pm 42 \mu\text{Gy}\cdot\text{a}^{-1}$  was thus used for the age calculation, but  
318 could be unrepresentative of the dose rate underwent by the samples unearthed from this GH  
319 (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  $106 \pm 23 \mu\text{Gy}\cdot\text{a}^{-1}$  was computed from the  
321 values recorded in GHs 15 and 17. For GHs 18 and 21, only one dosimeter could be inserted  
322 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  $94 \pm$   
324 10 ka (base of the sequence, GH 21) to  $35 \pm 8$  ka (top of the sequence, GH 15). Dose response

325 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  
327 case, the EU-ESR age represents the minimum age, and the US-ESR age is thought to be  
328 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  
331 dentine, describing sublinear and recent uptakes, respectively. In contrast, for all other  
332 samples, EU, LU (linear uptake, Ikeya, 1982), RU and US-ESR ages are not distinguishable,  
333 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  $D_{\max}$  is 1500 Gy,  $D_{\max}/D_e$  values are high, included between 131 and 302 (SOM Table S2). To  
338 test the impact of high irradiation steps on the  $D_e$  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  $D_e$  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  $D_e$  values are correlated with low U-  
346 content and environmental dose rates. Indeed, gamma (plus beta, if applicable) dose rates  
347 from the sediment are very low, ranging from  $64 \pm 15$  (GH 21) to  $108 \pm 42 \mu\text{Gy}\cdot\text{a}^{-1}$  (GH 15).  
348 However, the cosmic dose rate is significant, varying from 38 to  $99 \mu\text{Gy}\cdot\text{a}^{-1}$  depending on  
349 sample location. It represents up to 47% (GK 75-426) of the dose rate. By contrast, the sum of



350 the dose rates from each tissue does not exceed 6%, at the exception of GK 57-2609, which  
351 rises to 13% (Fig. 8).

352

#### 353 **4. Discussion**

354 These ESR ages obtained for the lower part of the stratigraphic sequence provide new  
355 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  
358 calculated. However, these two ages do not overlap. Two hypotheses could explain the age  
359 difference: 1) the deposition time of this layer is in the order of 10–40 ka or 2) diagenetic  
360 processes during burial could have altered GK 57-2609, which has a higher U-content (around  
361 0.26 ppm) in the enamel than in the dentine. Indeed, enamel is less porous than dentine and  
362 cement and so the U-content is generally lower (e.g., Eggins et al., 2003; Kohn, 2008). In the  
363 case of GK 57-2609, the U-content of 0.26 ppm in the enamel is not well understood,  
364 especially if we consider the other dated samples, for which the U-content does not exceed  
365 0.03 ppm. However, this hypothesis requires further investigation to assess the preservation  
366 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  $83 \pm 9$   
368 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  
376 from delayed neutron counting (U) and neutron activation (Th, K; Richter et al., 2000) and  
377 ranged from 79 to  $151 \mu\text{Gy}\cdot\text{a}^{-1}$ . However, our beta dose rate (measured using laboratory  
378 gamma ray spectrometry) is lower, ranging from  $32 \pm 4$  to  $44 \pm 6 \mu\text{Gy}\cdot\text{a}^{-1}$ . While in gamma  
379 spectrometry bulk samples, which include small pebbles, rocks, bones, etc. are analyzed, the  
380 small sample for NAA consisted of clayey sediment only. This was taken as a more realistic  
381 scenario by Richter et al. (2000) considering the short ranged beta contribution (around 2 mm)  
382 and that teeth are usually encased by clayey sediments. It could explain why the beta dose  
383 rates measured by Richter et al. (2000) are much higher than those we measured using  
384 laboratory gamma ray spectrometry. Moreover, cave deposits are generally heterogeneous  
385 dosimetric environments, due to the presence of limestone rocks for instance. Lateral  
386 variation of the gamma dose rate should be also considered as a potential source for the age  
387 discrepancy. Indeed, a value of  $67 \mu\text{Gy}\cdot\text{a}^{-1}$  was measured in GH 18 for the present study,  
388 while previous published ages by Richter et al. (2000) were calculated using a value of  $39$   
389  $\mu\text{Gy}\cdot\text{a}^{-1}$ . These results suggest that Neanderthals inhabited the cave not only at the beginning  
390 of MIS 3 but during MIS 4 as well. These ages, obtained for the lower part of the sequence,  
391 are systematically older than  $^{14}\text{C}$  ages. Their antiquity, combined with the fact that  $^{14}\text{C}$  ages  
392 obtained previously do not increase as a function of depth, indicate an occupation time  
393 beyond the limit of radiocarbon, i.e., older than 50 ka (Fig. 10). De facto, uncalibrated  $^{14}\text{C}$  age  
394 published by Higham et al. (2012) for GH 21, of  $48.6 \pm 3.2$  ka, should be considered as a  
395 minimum age. The two sets of results obtained for GHs 21 and 18 allow, for the first time, to  
396 constrain Neanderthal occupation at Geißenklösterle Cave and demonstrate the antiquity of  
397 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  $44$   
399  $\pm 5$  ka, and are in agreement with  $^{14}\text{C}$  ages (Fig. 10), except for the age published by Conard

400 and Bolus (2003). This date was not obtained using the ultrafiltration protocol, used to  
401 remove modern contaminants, which could explain why it appears younger than those  
402 published by Conard and Bolus (2008) and Higham et al. (2012). The fact that this unit is  
403 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 ( $46 \pm 3$  ka) coincides with Greenland  
405 Stadial (GS) 12 or 13. Stratigraphic discontinuities between Middle and Upper Paleolithic  
406 layers have also been identified elsewhere in Europe (e.g., Spain: Mallol et al., 2012; Italy:  
407 Peresani, 2008). This supports the hypothesis of a regional extinction or substantial  
408 depopulation of Neanderthals in the Swabian Jura prior to the arrival of modern humans  
409 (Conard et al. 2006). Radiocarbon ages indicate that early Aurignacian deposits predate  
410 Heinrich Stadial 4 (around 40–38 ka; Higham et al., 2012). Taking into account all available  
411 chronological data, GH 17 could have been deposited during Heinrich Stadial 5, which took  
412 place between ca. 49–47 ka (Svensson et al., 2008).

413 For the Aurignacian layers (GHs 15 and 16, AHs III, IIIa and IIIb), ESR ages are in  
414 agreement with  $^{14}\text{C}$  and TL data (Fig. 10). Three ages are available for GH 15, of  $40 \pm 10$  ka,  
415  $38 \pm 8$  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  
419 between these two ages could be explained by their stratigraphic position or by the spatial  
420 variation of the in situ gamma dose rate, which cannot be directly measured in GH 16. The  
421 youngest is attributed to a tooth sampled at a depth of 3.43 m, whereas the oldest age is  
422 associated with a sample found at a depth of 3.70 m (Fig. 9). However, since this layer was  
423 entirely excavated, the gamma dose rate cannot be measured in situ and no sediment was  
424 available for laboratory gamma ray spectrometry. It was thus not possible to assess the degree

425 of heterogeneity for GH 16. Considering that the ages for this layer were computed using the  
426 gamma dose rate recorded in both GHs 15 and 17, we do not exclude a local variation of the  
427 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  
429 may be obtained for the lower Aurignacian layers.

430

## 431 **6. Conclusions**

432 ESR dating was used to date herbivorous teeth from Middle Paleolithic and early  
433 Aurignacian horizons in order to extend the chronological framework of the Geißenklösterle  
434 sequence (Swabian Jura, Germany). The ESR results were compared with previous  $^{14}\text{C}$ , ESR  
435 and TL results available for the Aurignacian and the uppermost Middle Paleolithic layers.  
436 They demonstrate the potential and limitations of such application in a heterogeneous  
437 dosimetric environment. Indeed, the difficulty in reconstructing the gamma dose rate and  
438 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  $^{14}\text{C}$  and ESR  
440 ages obtained on GH 17 and early Aurignacian deposits bolsters the hypothesis of an early  
441 occupation by *H. sapiens* before 40 ka, and supports ESR ages obtained from Middle  
442 Paleolithic layers, which gives an age estimate for the onset of occupation in the cave, starting  
443 from MIS 5.

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

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

455 In the context of the end of the Middle Paleolithic, the study of Neanderthal  
456 occupations within a chronological framework at Geißenklösterle cave is fundamental to  
457 assess the spatiotemporal disappearance of this population from Europe, as well as to  
458 understand group dynamics during the Late Pleistocene. ESR ages obtained in this study  
459 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  
461 base of the sequence.

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

474

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484

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697

## 698 **Figure captions**

699

700 **Figure 1.** Location of Geißenklösterle (red dot) and other Late Pleistocene sites (black dot) in  
701 the Ach and Lone Valleys, Swabian Jura (Baden-Württemberg, Germany). Redrawn from  
702 Figure 19.1. in Conard (2011).

703

704 **Figure 2.** Stratigraphic column, teeth locations (white stars) and weighted mean ages obtained  
705 at Geißenklösterle. M: Magdalenian; G: Gravettian; UA: upper Aurignacian; LA: lower  
706 Aurignacian; OH: occupational hiatus; MP: Middle Palaeolithic. Redrawn from profile E  
707 from Figure 40 in Miller (2015).

708

709 **Figure 3.** Lithic artifacts from early Aurignacian layers. Reproduced from Hahn (1988).

710

711 **Figure 4.** Lithic artifacts from Late Middle Paleolithic layers. 1, 2: sidescrapers; 3: retouched  
712 flake with faceted platform remnant. Reproduced from Conard et al. (2006).

713

714 **Figure 5.** Map of Geißenklösterle (top right) with measured (red) and assumed (blue) roof  
715 thickness and photographs of the sections showing both dosimeters and sediment samples  
716 locations.

717

718 **Figure 6.** Cross section of the cave and 3D cosmic dose modelling. The cosmic dose field can  
719 be modelled as a cone with an angle of 30° in relation to the vertical axis, providing around  
720 65% of the cosmic dose rate, while cones with angles between 30° and 60° and 60° and 90°  
721 provide around 30% and 5% of the cosmic dose rate, respectively. Translated from Figure 5 in  
722 Richard et al. (2017).

723

724 **Figure 7.** ESR dose response curves of samples GK 76-1080, GH 15 (a) and GK 47-583, GH  
725 21 (b).

726

727 **Figure 8.** Relative contributions of alpha, beta, gamma and cosmic dose rates (%) to the total  
728 dose rate calculated for the ages presented in Table 5.

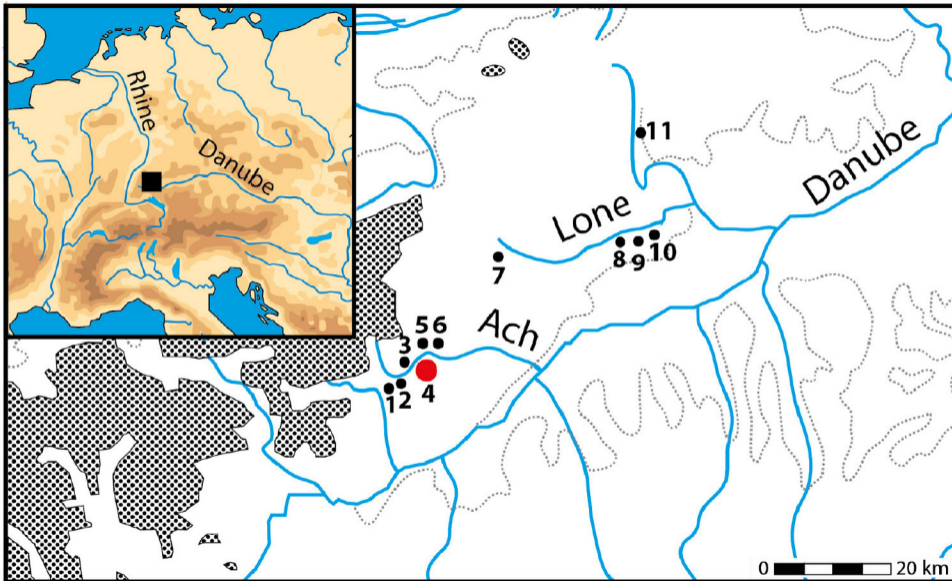
729

730 **Figure 9.** Graphical representation of ages as a function of the depth and the  $\delta^{18}\text{O}$  variations  
731 (North Greenland Ice Core Project (NGRIP) dataset (Rasmussen et al., 2014; Seierstad et al.,  
732 2014), available on [www.iceandclimate.dk/data](http://www.iceandclimate.dk/data)).

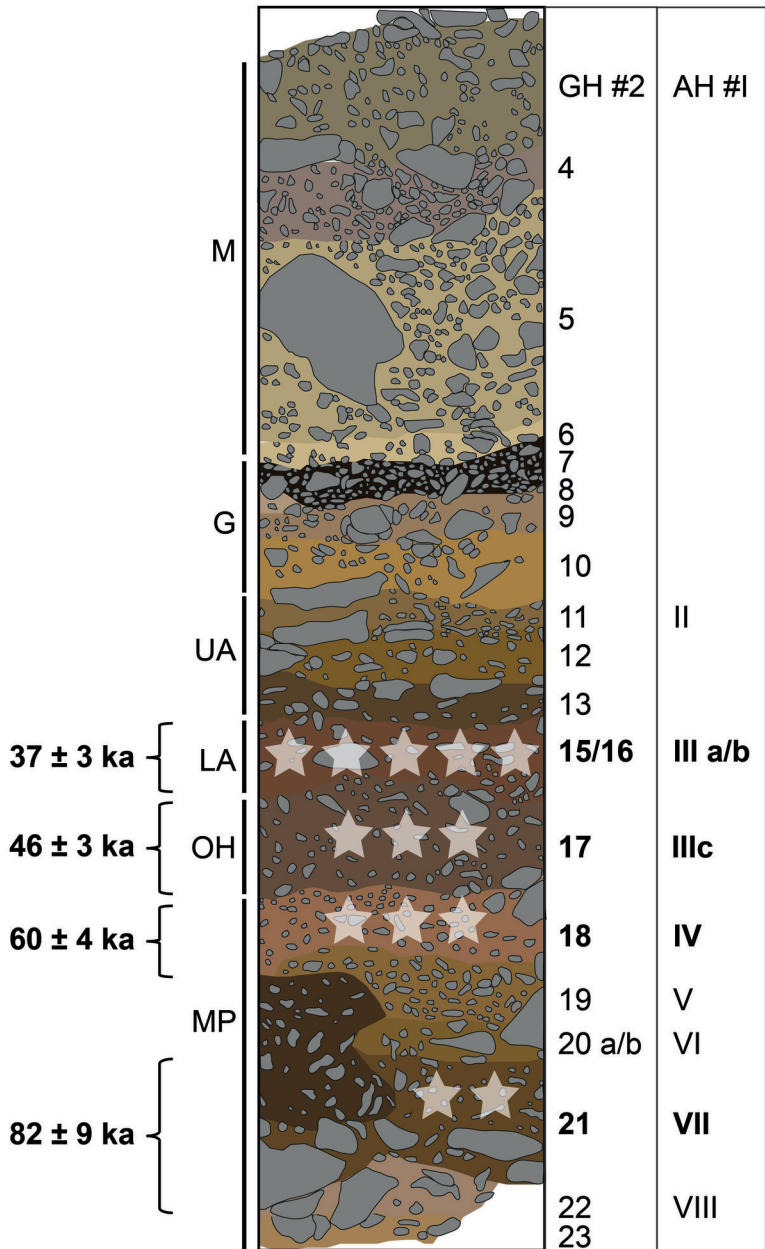
733

734 **Figure 10.** Comparison of published  $^{14}\text{C}$ , TL and ESR ages ( $1\sigma$ ) with ESR ages obtained in  
735 this study.  $^{14}\text{C}$  ages are calibrated using IntCal13 (Reimer et al., 2013) and Oxcal 4.2 (Bronk  
736 Ramsey and Lee, 2013).





- 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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2



3





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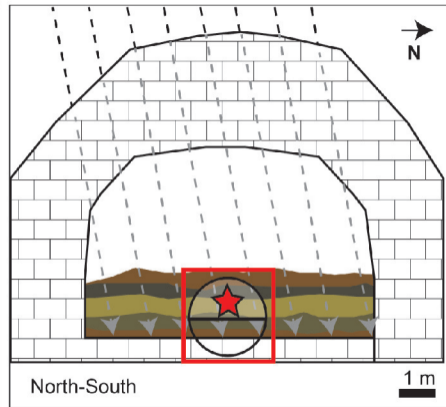
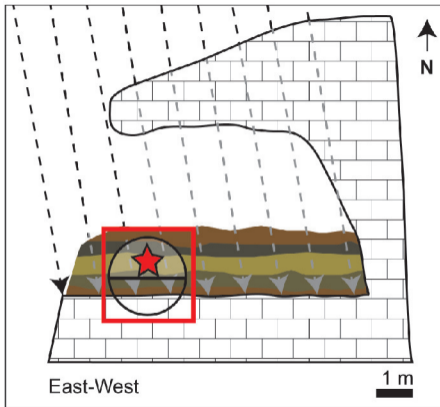
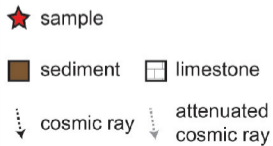
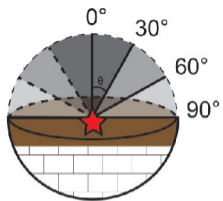


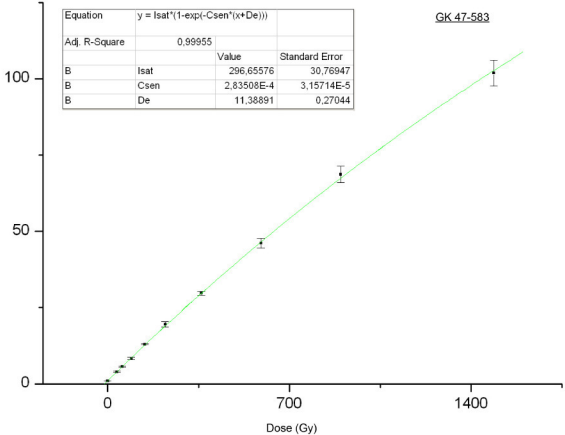
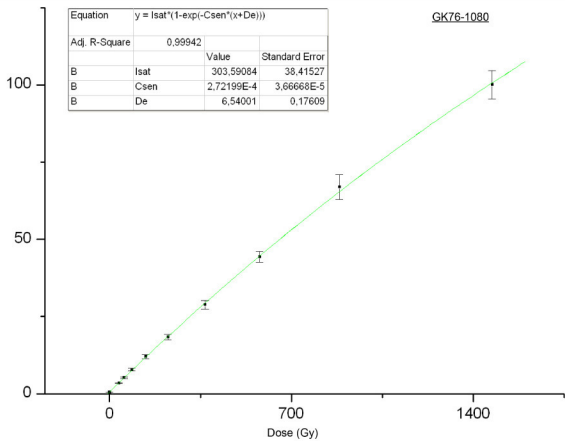
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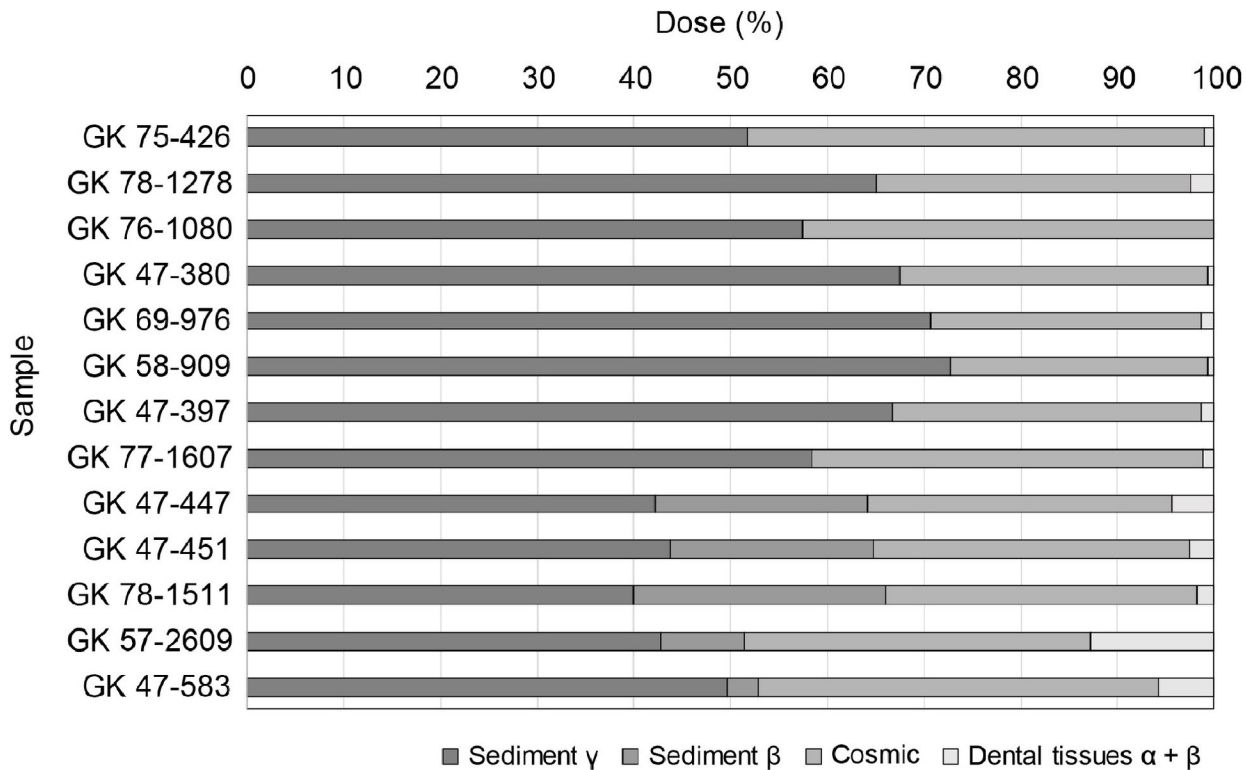
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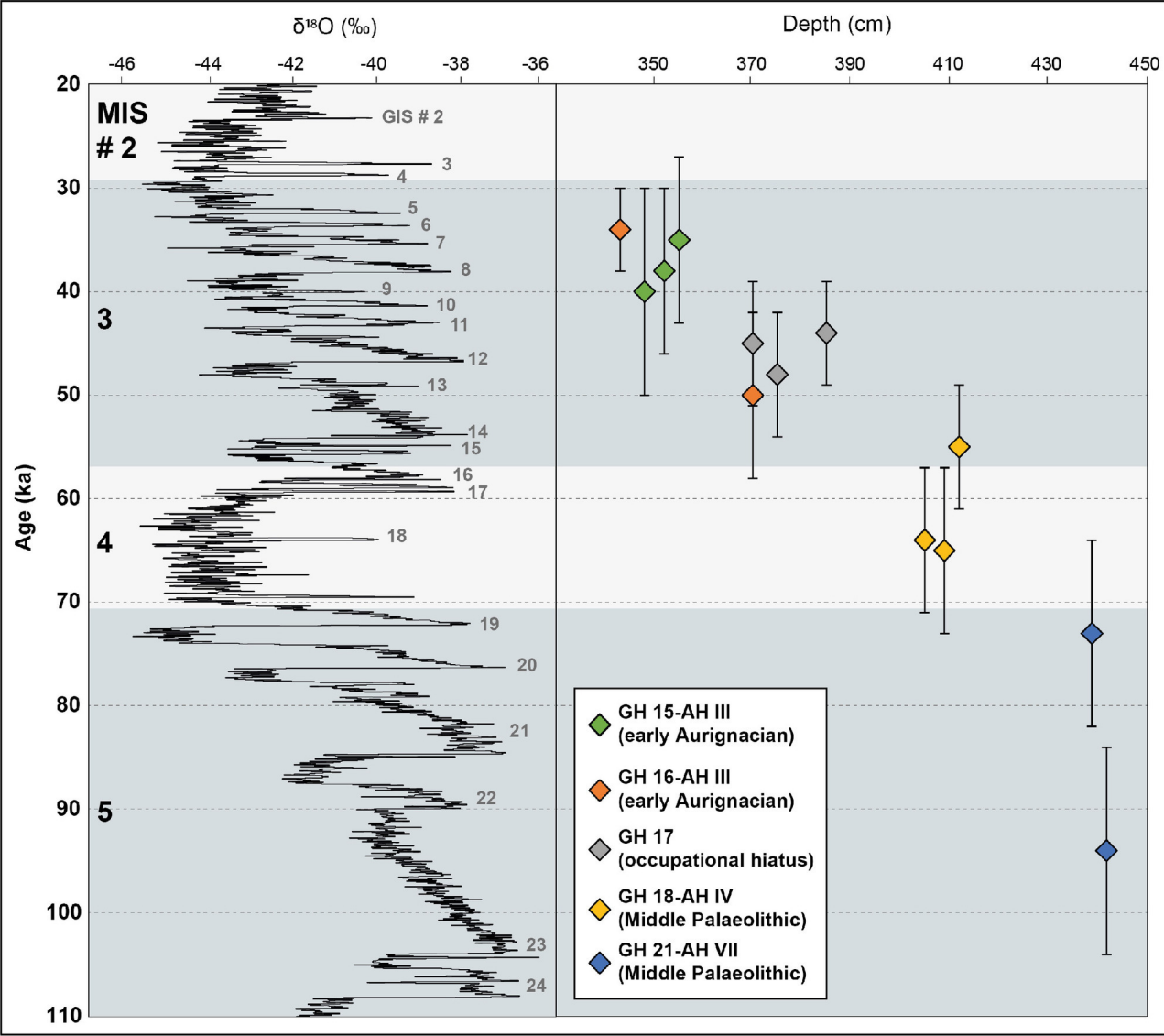


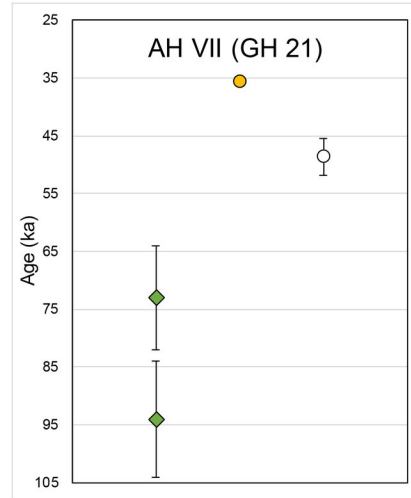
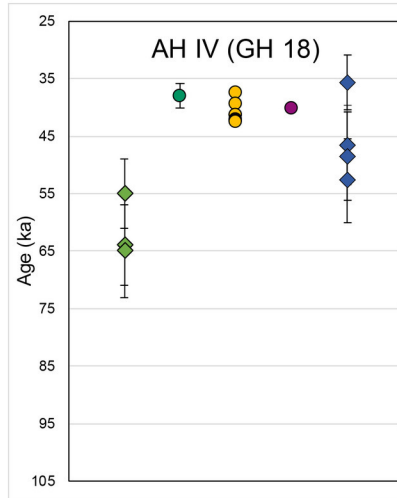
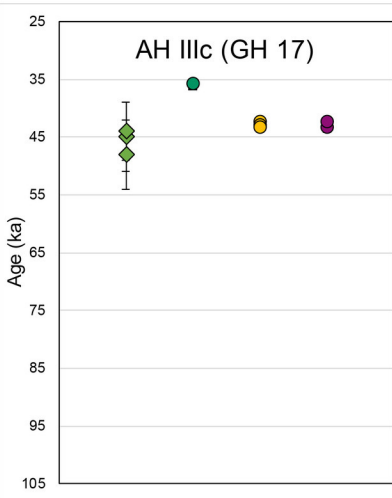
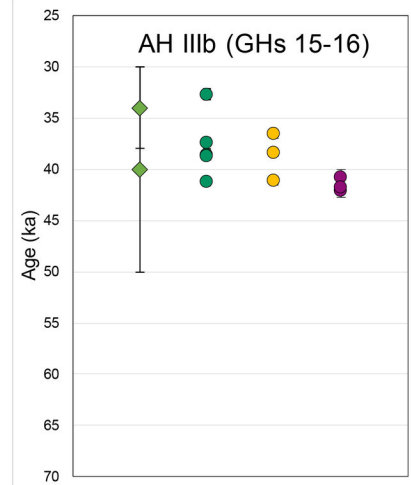
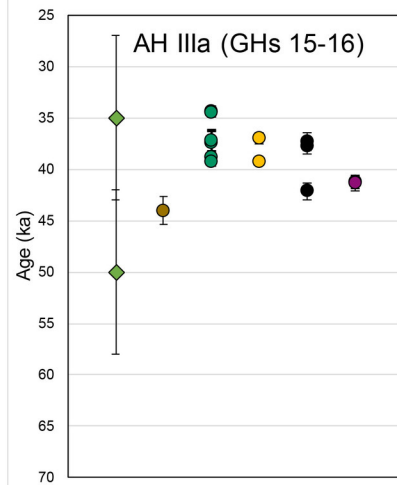
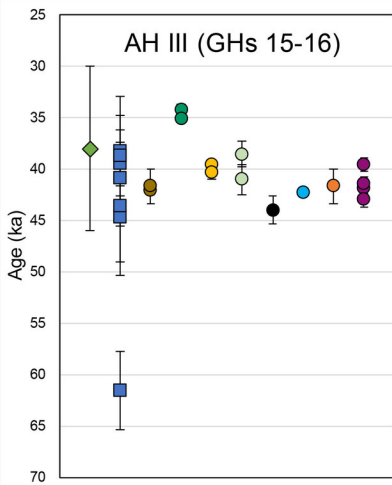












- <sup>14</sup>C cal BP (Richter et al., 2000)
- <sup>14</sup>C cal BP (Hofreiter et al., 2007)
- <sup>14</sup>C cal BP (Housley et al., 1997)
- <sup>14</sup>C cal BP (Hahn, 1982)
- <sup>14</sup>C cal BP (Conard and Bolus, 2008)
- <sup>14</sup>C cal BP (Hahn, 1995)
- <sup>14</sup>C cal BP (Higham et al., 2012)
- <sup>14</sup>C cal BP (Conard and Bolus, 2003)
- <sup>14</sup>C uncal BP (Higham et al., 2012)
- TL (Richter et al., 2000)
- ◆ ESR (Richter et al., 2000)
- ◆ ESR (this work)

**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°      | GH | AH   | Square | X (m) | Y (m) | Z (m) | Taxon                 | Grain size (µm) <sup>a</sup> |
|----------|-------------|----|------|--------|-------|-------|-------|-----------------------|------------------------------|
| Tooth    | GK 75-426   | 15 | III  | 75     | 25.57 | 29.13 | -3.52 | <i>Equus</i> sp.      | 100–200                      |
| Tooth    | GK 78-1278  | 15 | IIIb | 78     | 28.49 | 29.85 | -3.48 | <i>Equus</i> sp.      | 100–200                      |
| Tooth    | GK 76-1080  | 15 | IIIa | 76     | 26.59 | 29.72 | -3.55 | <i>Equus</i> sp.      | 0–200                        |
| Sediment | GK.sed.GH15 | 15 | III  | 99     | 29.21 | 31.12 | -3.30 | —                     | —                            |
| Tooth    | GK 47-380   | 16 | IIIa | 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 | IIIc | 58     | 28.19 | 27.85 | -3.70 | <i>Equus</i> sp.      | 100–200                      |
| Tooth    | GK 47-397   | 17 | IIIc | 47     | 27.42 | 26.57 | -3.75 | <i>Equus</i> sp.      | 100–200                      |
| Tooth    | GK 77-1607  | 17 | IIIc | 77     | 27.05 | 29.06 | -3.85 | <i>Equus</i> sp.      | 100200                       |
| Sediment | GK.sed.GH17 | 17 | IIIc | 69     | 29.17 | 28.37 | -3.82 | —                     | —                            |
| Tooth    | GK 47-447   | 18 | IV   | 47     | 27.06 | 26.96 | -4.12 | <i>Capra ibex</i>     | 0–200                        |
| Tooth    | GK 47-451   | 18 | IV   | 47     | 27.44 | 26.43 | -4.05 | <i>Capra ibex</i>     | 100–200                      |
| Tooth    | GK 78-1511  | 18 | IV   | 78     | 28.50 | 29.05 | -4.09 | <i>Capra ibex</i>     | 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 | —                     | —                            |

<sup>a</sup> 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).

| Sample     | GH | Tissue         | [ <sup>238</sup> U] (ppm) | <sup>234</sup> U/ <sup>238</sup> U | <sup>230</sup> Th/ <sup>234</sup> U | Enamel (initial thickness, μm) | Side 1 (removed thickness, μm) | Side 2 (removed thickness, μm) |
|------------|----|----------------|---------------------------|------------------------------------|-------------------------------------|--------------------------------|--------------------------------|--------------------------------|
| GK 75-426  | 15 | E              | 0.0024 ± 0.0001           | nd                                 | nd                                  | 1201 ± 120                     | 88 ± 9                         | 67 ± 7                         |
|            |    | D              | 0.0213 ± 0.0001           | 1.0872 ± 0.0242                    | 0.8249 ± 0.2152                     |                                |                                |                                |
|            |    | C              | 0.2336 ± 0.0013           | 1.2043 ± 0.0057                    | 0.1111 ± 0.0080                     |                                |                                |                                |
| GK 78-1278 | 15 | E              | 0.0027 ± 0.0001           | 1.3579 ± 0.0418                    | nd                                  | 1177 ± 118                     | 128 ± 13                       | 54 ± 5                         |
|            |    | D              | 0.1040 ± 0.0006           | 1.3526 ± 0.0231                    | 0.6644 ± 0.0171                     |                                |                                |                                |
|            |    | C              | 0.3650 ± 0.0025           | 1.1613 ± 0.0130                    | 0.1246 ± 0.0037                     |                                |                                |                                |
| GK 76-1080 | 15 | E              | 0.0041 ± 0.0001           | 1.5452 ± 0.0570                    | nd                                  | 1137 ± 114                     | 99 ± 10                        | 169 ± 17                       |
|            |    | D <sup>a</sup> | 0.0314 ± 0.022            | nd                                 | nd                                  |                                |                                |                                |
|            |    | C              | 0.0492 ± 0.0004           | 1.1855 ± 0.0481                    | 0.3468 ± 0.0201                     |                                |                                |                                |
| GK 47-380  | 16 | E              | 0.0033 ± 0.0001           | 1.3840 ± 0.3685                    | nd                                  | 1190 ± 119                     | 106 ± 11                       | 105 ± 11                       |
|            |    | D <sup>a</sup> | 0.0346 ± 0.0143           | nd                                 | nd                                  |                                |                                |                                |
|            |    | C <sup>a</sup> | 0.1617 ± 0.0213           | nd                                 | nd                                  |                                |                                |                                |
| GK 69-976  | 16 | E              | 0.0037 ± 0.0001           | 1.6266 ± 0.4542                    | nd                                  | 1161 ± 116                     | 119 ± 12                       | 61 ± 6                         |
|            |    | D <sup>a</sup> | 0.0376 ± 0.0155           | nd                                 | nd                                  |                                |                                |                                |
|            |    | C <sup>a</sup> | 0.1797 ± 0.0327           | nd                                 | nd                                  |                                |                                |                                |
| GK 58-909  | 17 | E              | 0.0033 ± 0.0001           | 1.4217 ± 0.3458                    | nd                                  | 1374 ± 137                     | 77 ± 8                         | 74 ± 7                         |
|            |    | D              | 0.0120 ± 0.0001           | 1.3358 ± 0.0274                    | nd                                  |                                |                                |                                |
|            |    | C              | 0.0969 ± 0.0007           | 1.2396 ± 0.0134                    | 0.1231 ± 0.0068                     |                                |                                |                                |
| GK 47-397  | 17 | E              | 0.0026 ± 0.0001           | 1.6419 ± 0.7889                    | nd                                  | 1238 ± 124                     | 117 ± 12                       | 77 ± 8                         |
|            |    | D              | 0.0319 ± 0.0002           | 1.2473 ± 0.0277                    | 0.4596 ± 0.0462                     |                                |                                |                                |
|            |    | C              | 0.2904 ± 0.0018           | 1.2474 ± 0.0201                    | 0.1915 ± 0.0055                     |                                |                                |                                |
| GK 77-1607 | 17 | E              | 0.0082 ± 0.0001           | 1.4501 ± 0.0149                    | nd                                  | 1288 ± 129                     | 116 ± 12                       | 22 ± 2                         |
|            |    | D              | 0.0984 ± 0.0014           | 1.2987 ± 0.1183                    | 0.1750 ± 0.0285                     |                                |                                |                                |
|            |    | C              | 0.0364 ± 0.0002           | nd                                 | nd                                  |                                |                                |                                |
| GK 47-447  | 18 | E              | 0.0294 ± 0.0001           | 1.2475 ± 0.0207                    | 0.2614 ± 0.0291                     | 821 ± 82                       | 37 ± 4                         | 25 ± 3                         |
|            |    | D              | 0.0627 ± 0.0004           | 1.2751 ± 0.0102                    | 0.3601 ± 0.0168                     |                                |                                |                                |
| GK 47-451  | 18 | E              | 0.0108 ± 0.0001           | 1.8344 ± 0.3333                    | nd                                  | 847 ± 45                       | 78 ± 8                         | 67 ± 7                         |
|            |    | D              | 0.0390 ± 0.0002           | 1.2282 ± 0.0611                    | 0.1971 ± 0.0203                     |                                |                                |                                |
| GK 78-1511 | 18 | E              | 0.0106 ± 0.0001           | 1.2740 ± 0.0410                    | nd                                  | 698 ± 70                       | 149 ± 15                       | 31 ± 3                         |
|            |    | D              | 0.0541 ± 0.0003           | 1.2413 ± 0.0637                    | 0.2113 ± 0.0098                     |                                |                                |                                |
| GK 57-2609 | 21 | E              | 0.2583 ± 0.0016           | 1.3632 ± 0.0124                    | 0.2018 ± 0.0079                     | 1057 ± 106                     | 211 ± 21                       | 24 ± 2                         |
|            |    | D              | 0.0556 ± 0.0001           | 1.2319 ± 0.0342                    | 0.1272 ± 0.0074                     |                                |                                |                                |
| GK 47-583  | 21 | E              | 0.0264 ± 0.0001           | 1.3225 ± 0.0590                    | nd                                  | 2346 ± 235                     | 87 ± 9                         | 95 ± 9                         |

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

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Abbreviations: nd = not detectable; E = enamel; 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**

$^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  measured using a HPGe gamma spectrometer. The position of each sediment sample is available in Figure 5.

| Sample      | GH | $^{238}\text{U}$ (ppm) | $^{232}\text{Th}$ (ppm) | $^{40}\text{K}$ (%) |
|-------------|----|------------------------|-------------------------|---------------------|
| 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 | $0.20 \pm 0.05$        | $0.81 \pm 0.06$         | $0.06 \pm 0.01$     |

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).

| GH | No. | Section | x (m)  | y (m)  | z (m) | $\gamma$ dose rate ( $\mu\text{Gy/a}$ ) | Mean value ( $\mu\text{Gy/a}$ ) |
|----|-----|---------|--------|--------|-------|---|---------------------------------|
| 15 | 1   | G       | 28.789 | 31.122 | -3.30 | 145                                     | 108 $\pm$ 42                    |
| 15 | 2   | G       | 29.632 | 31.086 | -3.34 | 66                                      |                                 |
| 15 | 3   | G       | 30.434 | 31.047 | -3.34 | 113                                     |                                 |
| 16 | —   | —       | —      | —      | —     | —                                       | 106 $\pm$ 23 <sup>a</sup>       |
| 17 | 4   | B       | 29.168 | 28.173 | -3.92 | 88                                      | 104 $\pm$ 18                    |
| 17 | 5   | B       | 29.182 | 28.62  | -3.68 | 103                                     |                                 |
| 17 | 7   | E       | 28.477 | 26.413 | -3.79 | 122                                     |                                 |
| 18 | 8   | B       | 29.184 | 28.587 | -4.08 | 67                                      | 67 $\pm$ 17 <sup>b</sup>        |
| 21 | 12  | B       | 29.134 | 27.977 | -4.48 | 60                                      | 60 $\pm$ 15 <sup>b</sup>        |

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.

| Sample     | GH | D <sub>e</sub> (Gy) | Dose rate (μGy/a) |        |                     |                  | Age (ka) |                           |
|------------|----|---------------------|-------------------|--------|---------------------|------------------|----------|---------------------------|
|            |    |                     | Sediment<br>(γ)   | (β)    | Cosmic <sup>a</sup> | Tooth<br>(α + β) |          | Total<br>(α + β + γ)      |
| GK 75-426  | 15 | 7.92 ± 0.24         | 108 ± 42          | —      | 99 ± 10             | 2 ± 1            | 209 ± 41 | 38 ± 8                    |
| GK 78-1278 |    | 6.63 ± 0.16         | 108 ± 42          | —      | 54 ± 5              | 4 ± 1            | 166 ± 40 | 40 ± 10                   |
| GK 76-1080 |    | 6.54 ± 0.18         | 108 ± 42          | —      | 80 ± 8              | —                | 188 ± 40 | 35 ± 8                    |
| GK 47-380  | 16 | 7.75 ± 0.31         | 106 ± 23          | —      | 50 ± 5              | 1 ± 1            | 157 ± 22 | 50 ± 8                    |
| GK 69-976  |    | 4.94 ± 0.18         | 106 ± 23          | —      | 42 ± 4              | 2 ± 2            | 150 ± 22 | 34 ± 4                    |
| GK 58-909  | 17 | 6.42 ± 0.29         | 104 ± 18          | —      | 38 ± 4              | 1 ± 1            | 143 ± 18 | 45 ± 6                    |
| GK 47-397  |    | 7.56 ± 0.20         | 104 ± 18          | —      | 50 ± 5              | 2 ± 1            | 156 ± 18 | 48 ± 6                    |
| GK 77-1607 |    | 7.87 ± 0.23         | 104 ± 18          | —      | 72 ± 7              | 2 ± 1            | 178 ± 19 | 44 ± 5                    |
| GK 47-447  | 18 | 8.69 ± 0.24         | 67 ± 17           | 35 ± 4 | 50 ± 5              | 7 ± 1            | 159 ± 15 | 55 ± 6                    |
| GK 47-451  |    | 9.74 ± 0.24         | 67 ± 17           | 32 ± 4 | 50 ± 5              | 4 ± 2            | 153 ± 15 | 64 ± 7                    |
| GK 78-1511 |    | 10.92 ± 0.36        | 67 ± 17           | 44 ± 6 | 54 ± 5              | 3 ± 1            | 168 ± 15 | 65 ± 8                    |
| GK 57-2609 | 21 | 10.15 ± 0.46        | 60 ± 15           | 12 ± 2 | 50 ± 5              | 18 ± 5           | 140 ± 14 | <b>73 ± 9<sup>b</sup></b> |
| GK 47-583  |    | 11.39 ± 0.27        | 60 ± 15           | 4 ± 1  | 50 ± 5              | 7 ± 1            | 121 ± 13 | 94 ± 10                   |

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 ± 0.40 (enamel) and 2.15 ± 0.17 (dentine).