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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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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 ± 10 ka (GH 21) and 55 ± 6 ka (GH 18) by ESR on tooth enamel. Mean
44 ages for GH 17, at 46 ± 3 ka, and for the lower Aurignacian layers, at 37 ± 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}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.,
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, therianthropic
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}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 ± 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 ± 7 ka to 36 ± 5 ka (mean age = 43.3 ± 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}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}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}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 ¹⁴C
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 μm 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 μm granulometric
204 fraction was used for ESR measurements. For powder samples weighing less than 500 mg, the
205 0–100 and 100–200 μm 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}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}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}U , ^{232}Th and ^{40}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}U , ^{232}Th ,
255 and ^{40}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 σ 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).

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}U
292 and ^{230}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 ± 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}U , ^{232}Th and ^{40}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 ± 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 ± 6 ka) and the US-ESR (73 ± 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 (≈ 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 ± 0.40 for the enamel and 2.15 ± 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 ± 0.2 to 10.9 ± 0.4 , with
344 those published by Richter et al. (2000), ranging from 8.1 ± 0.7 to 10 ± 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 ± 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 ± 10 ka (GK 47-583) and 73 ± 9 ka (GK 57-
357 2609), indicate an occupation during the MIS 5, and a weighted mean age of 82 ± 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 ± 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 ± 10 ka (EU) and 100 ± 12 ka (RU).

370 For GH 18 (AH IV), the new ESR ages, ranging from 65 ± 8 ka to 55 ± 6 ka, are
371 somewhat older than previous ESR ages (Richter et al., 2000; from 53 ± 5 ka to 36 ± 5 ka;
372 Fig. 10). The weighted mean age is 60 ± 4 ka, while the one published by Richter et al. (2000)
373 is 43 ± 4 ka. The equivalent doses reported in Richter et al. (2000) for the dated teeth range
374 from 8.1 ± 0.7 to 10 ± 0.6 Gy and are similar to those obtained in the present study, ranging

375 from 8.7 ± 0.2 to 10.9 ± 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 ± 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}C ages. Their antiquity, combined with the fact that ^{14}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}C age
394 published by Higham et al. (2012) for GH 21, of 48.6 ± 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 ± 6 ka to 44
399 ± 5 ka, and are in agreement with ^{14}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 ± 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}C and TL data (Fig. 10). Three ages are available for GH 15, of 40 ± 10 ka,
415 38 ± 8 ka and 35 ± 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 ± 5 ka may be obtained.
417 These ages are constrained by the two other ages obtained for GH 16 (50 ± 8 ka and 34 ± 4 ;
418 Fig. 9), for which a weighted mean age of 37 ± 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 ± 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}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}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 ± 10 ka to 55 ± 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 ± 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}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

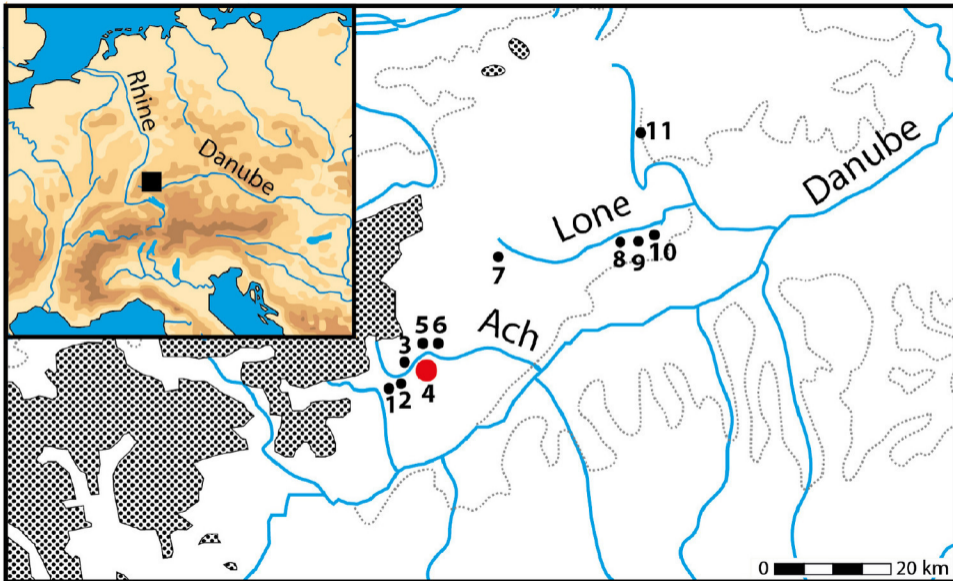
727 **Figure 8.** Relative contributions of alpha, beta, gamma and cosmic dose rates (%) to the total
728 dose rate calculated for the ages presented in Table 5.

729

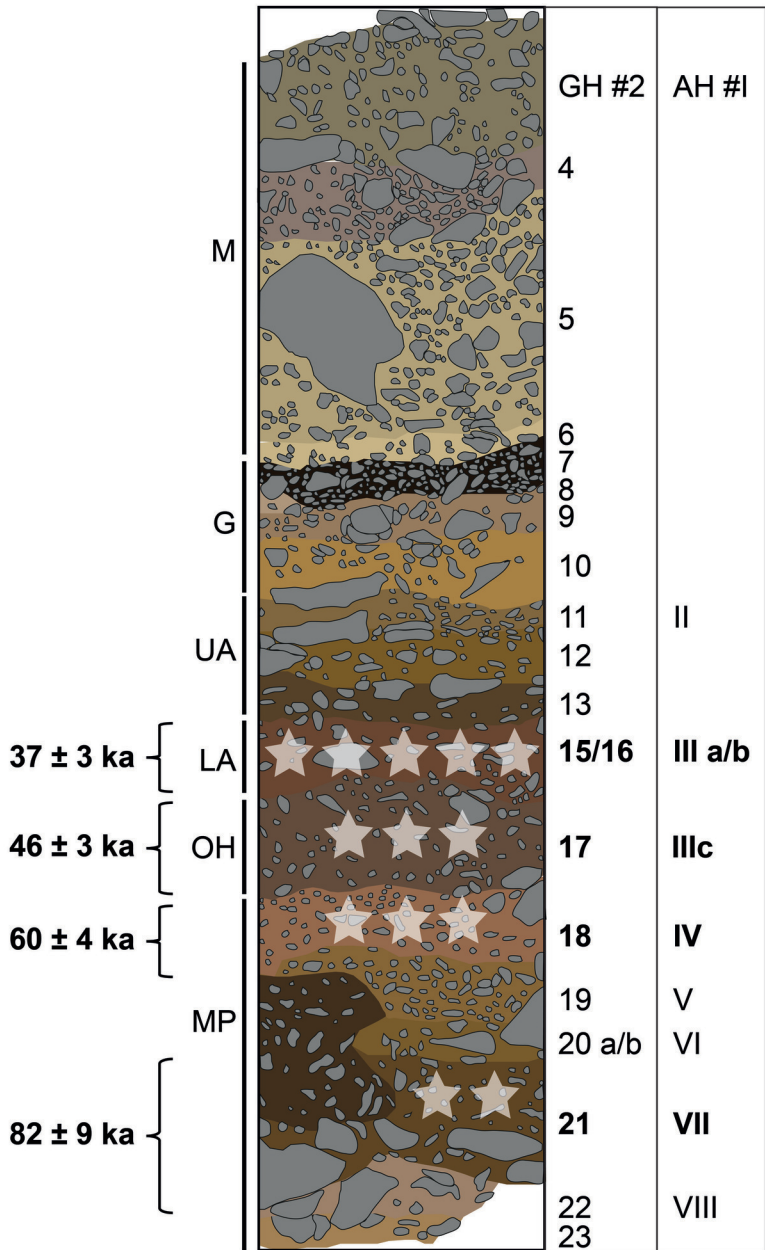
730 **Figure 9.** Graphical representation of ages as a function of the depth and the $\delta^{18}\text{O}$ variations
731 (North Greenland Ice Core Project (NGRIP) dataset (Rasmussen et al., 2014; Seierstad et al.,
732 2014), available on www.iceandclimate.dk/data).

733

734 **Figure 10.** Comparison of published ^{14}C , TL and ESR ages (1σ) with ESR ages obtained in
735 this study. ^{14}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





1



2



3





1



2



3



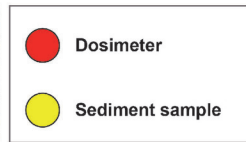
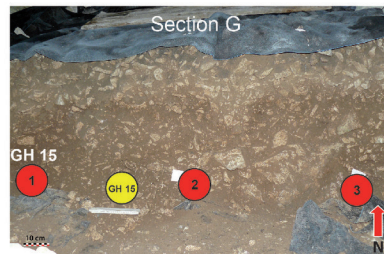
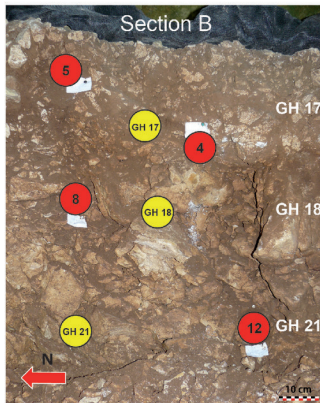
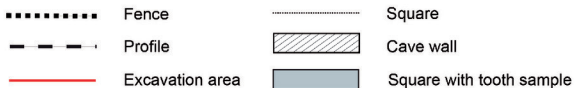
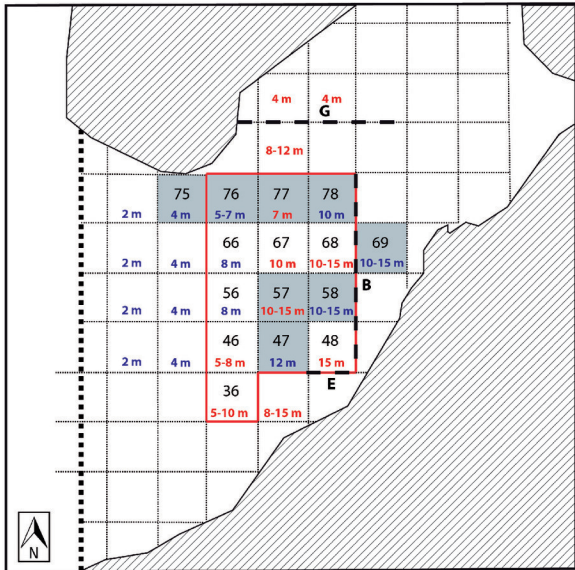
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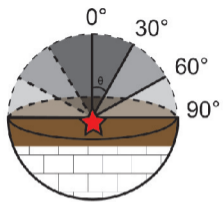


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5 cm



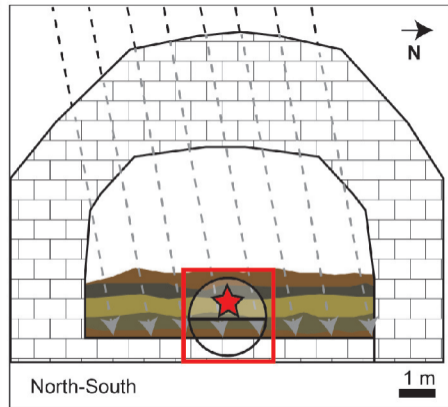
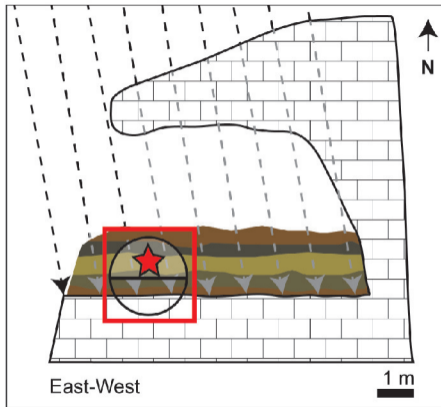


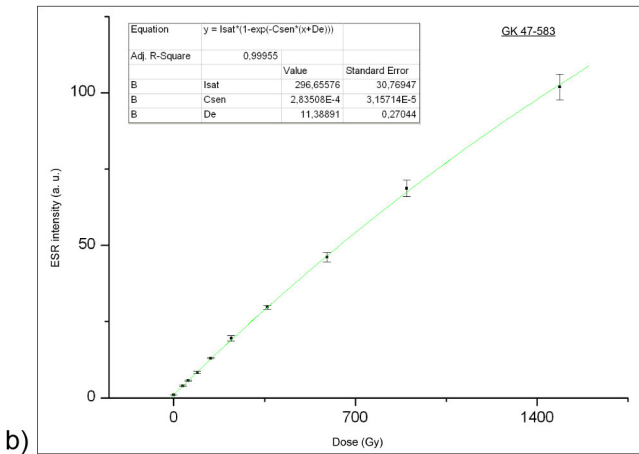
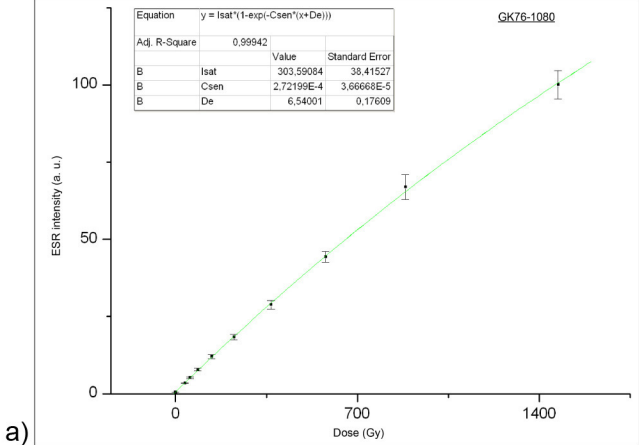


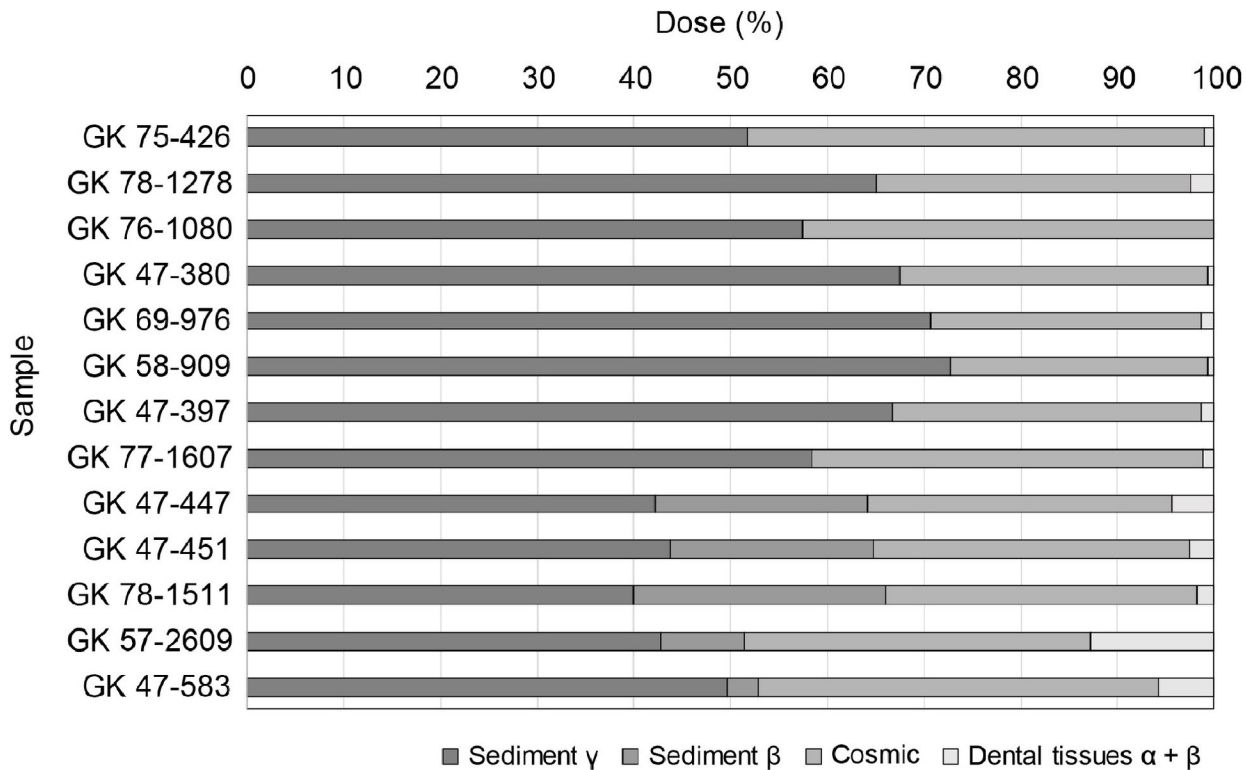
★ sample

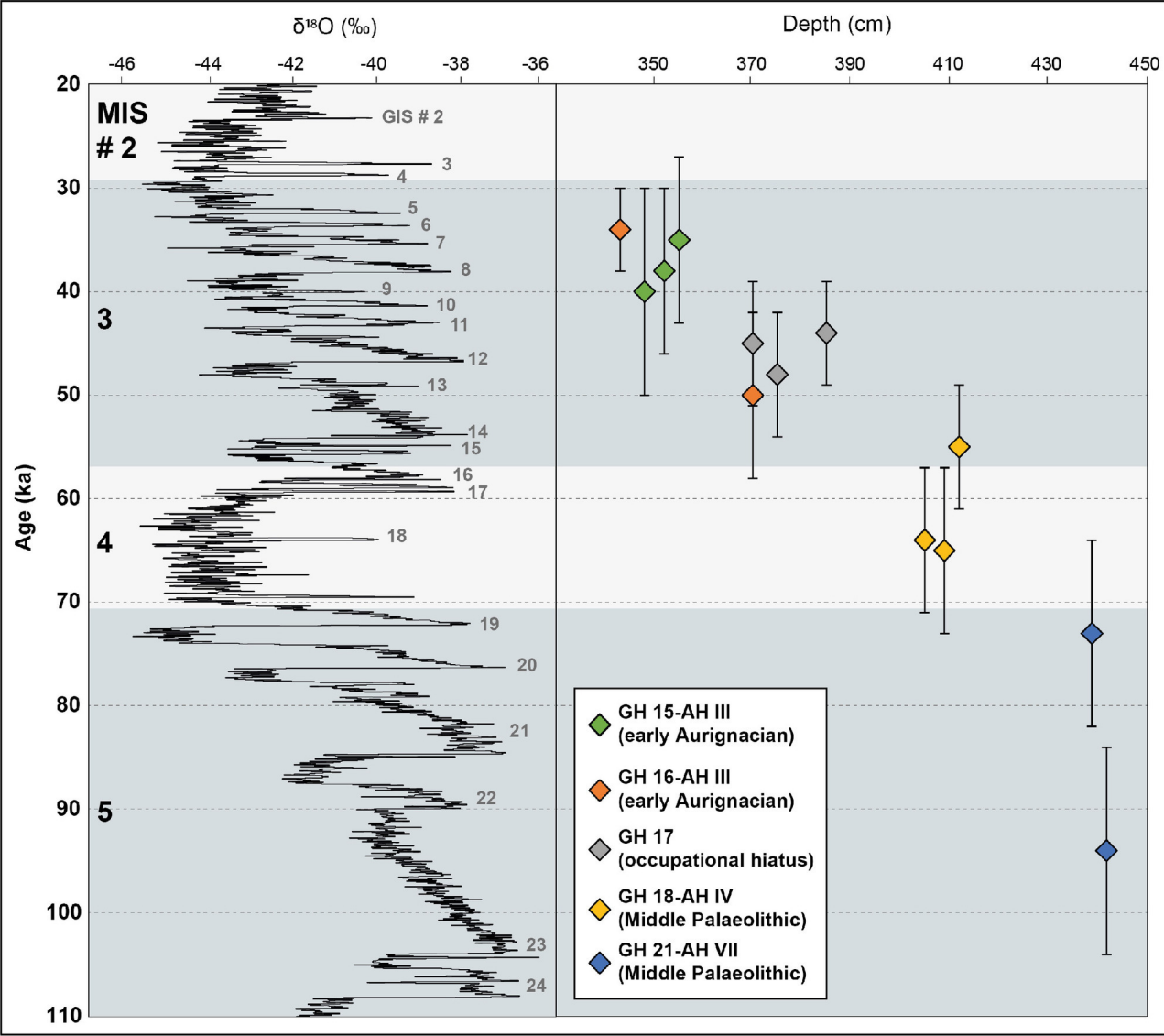
■ sediment □ limestone

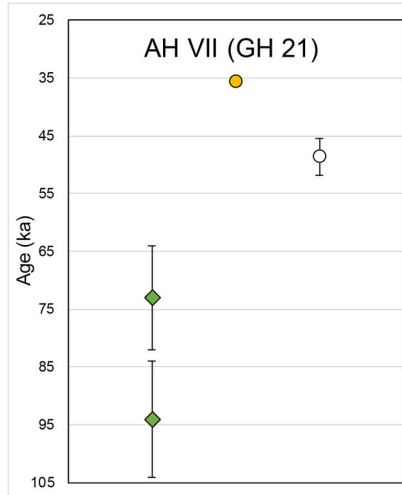
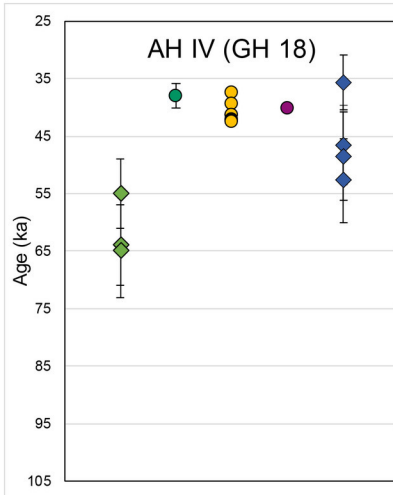
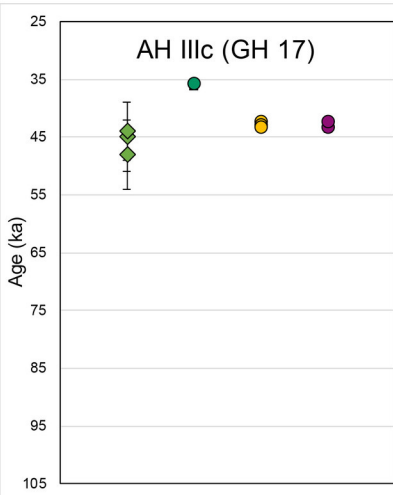
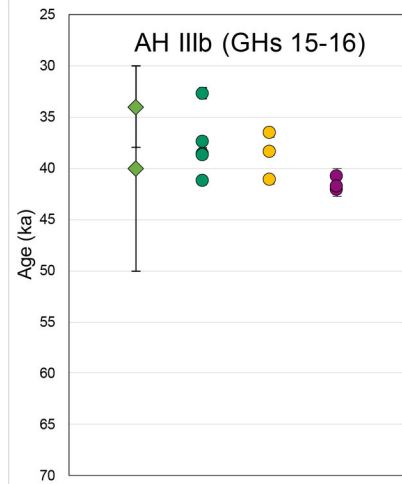
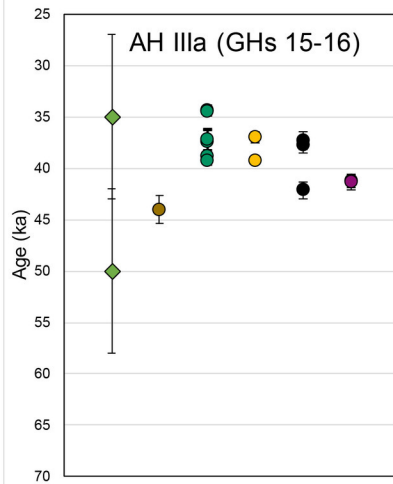
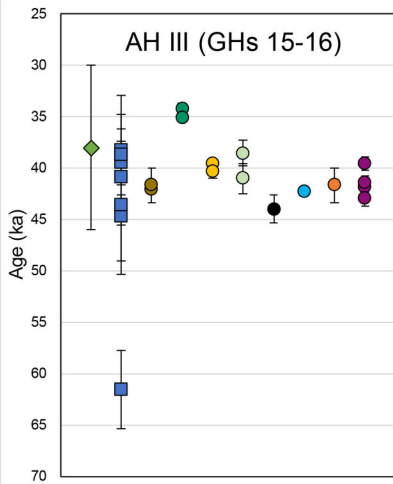
↘ cosmic ray ↘ attenuated cosmic ray











● ^{14}C cal BP (Richter et al., 2000)

● ^{14}C cal BP (Hahn, 1982)

● ^{14}C cal BP (Hahn, 1995)

● ^{14}C cal BP (Conard and Bolus, 2003)

● ^{14}C cal BP (Hofreiter et al., 2007)

● ^{14}C cal BP (Conard and Bolus, 2008)

● ^{14}C cal BP (Higham et al., 2012)

○ ^{14}C uncal BP (Higham et al., 2012)

● ^{14}C cal BP (Housley et al., 1997)

■ 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) ^a
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	—	—

^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).

Sample	GH	Tissue	[²³⁸ U] (ppm)	²³⁴ U/ ²³⁸ U	²³⁰ Th/ ²³⁴ 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 ^a	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 ^a	0.0346 ± 0.0143	nd	nd			
		C ^a	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 ^a	0.0376 ± 0.0155	nd	nd			
		C ^a	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 ± 0.0005 1.3057 ± 0.0185 0.2460 ± 0.0123

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

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

Table 3

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

Sample	GH	^{238}U (ppm)	^{232}Th (ppm)	^{40}K (%)
GK.sed.GH15	15	0.94 ± 0.07	2.89 ± 0.10	0.30 ± 0.01
GK.sed.GH17	17	0.54 ± 0.07	2.37 ± 0.01	0.22 ± 0.01
GK.sed.GH18	18	0.58 ± 0.07	1.59 ± 0.09	0.16 ± 0.01
GK.sed.GH21	21	0.20 ± 0.05	0.81 ± 0.06	0.06 ± 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)	γ 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 ^a
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 ^b
21	12	B	29.134	27.977	-4.48	60	60 \pm 15 ^b

Abbreviation: GH = geological horizon.

^aThis value corresponds to a mean value obtained from measurements in both GHs 15 and 17 (see text).

^bA 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 _e (Gy)	Dose rate (μGy/a)				Age (ka)	
			Sediment (γ)	(β)	Cosmic ^a	Tooth (α + β)		Total (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	73 ± 9^b
GK 47-583		11.39 ± 0.27	60 ± 15	4 ± 1	50 ± 5	7 ± 1	121 ± 13	94 ± 10

Abbreviation: GH = geological horizon.

^a A 10% error was estimated for the cosmic dose.^b US-ESR age with associated *p*-values of 0.80 ± 0.40 (enamel) and 2.15 ± 0.17 (dentine).