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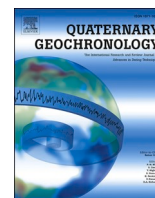
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## ESR/U-series dating of Eemian human occupations of Northern France

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### ABSTRACT

Palaeolithic sites associated with the Eemian Interglacial (MIS 5e) are very rare in NW Europe, and especially in Northern France, where their preservation is restricted to very specific geological contexts, in association with carbonated tufa (Caours) or peat deposits (Waziers). In order to check the reliability of ESR/U-series method to date teeth recovered from archaeological levels in such specific geological environments, teeth were sampled on these two Middle Palaeolithic sites and systematic *in situ* dosimetry was performed using portable gamma spectrometer. The ESR/U-series ages obtained on the Caours site are very homogeneous allowing the calculation of a mean age equal to  $125 \pm 11$  ka, in agreement with the geological age, mammal's association and other available geochronological data (U-series on tufa carbonate, TL on burnt flints, OSL on sediments), despite a relatively heterogeneous dosimetric environment (gamma dose rate ranging between ca 200–450  $\mu\text{Gy/a}$ ). At Waziers, reducing (water logging) environment linked to the peat leads to very specific U-series data of the analysed teeth (U content lower than 0.1 ppm in all the dental tissues, evidence of leaching in some tissues), but the mean ESR/U-series age,  $129 \pm 4$  ka, is also in agreement with the available geological and palaeoenvironmental data indicating that the dated teeth were associated with Late Glacial deposits prior to the climatic interglacial optimum. These two case studies then confirm the reliability of ESR/U-series method to date with good reliability and accuracy the archaeological levels linked to such relatively short climatic events (ca 10 ka).

### 1. Introduction

Since the late 1980s, ESR/U-series dating method has been used to date Middle Palaeolithic sites of Western Europe, alone or in combination with other geochronological methods. The method was particularly applied on teeth recovered from sites in Northern France, where the age of the archaeological levels is often well constrained by stratigraphical or paleoenvironmental data (e.g. Bahain et al., 2010, 2012, 2015, 2020; Antoine et al., 2016, 2021; Guérin et al., 2017). Despite a relatively poor

precision by comparison to  $^{14}\text{C}$  or OSL results, ESR/U-series data usually enable to correlate the dated levels to a given marine isotopic stage and participate hence to the construction of a unified regional chronostratigraphical framework (Bahain et al., 2007; Antoine et al., 2000, 2007, 2014; Voinchet et al., 2015). However, this poor precision can be a limitation to date levels associated to relatively short climatic or geological events, such as Late Pleistocene Greenland stadials and interstadials (Rasmussen et al., 2014) or the last interglacial stage (MIS 5e, ca 129–116 ka, Sakari Salonen et al., 2018), called Eemian in northern

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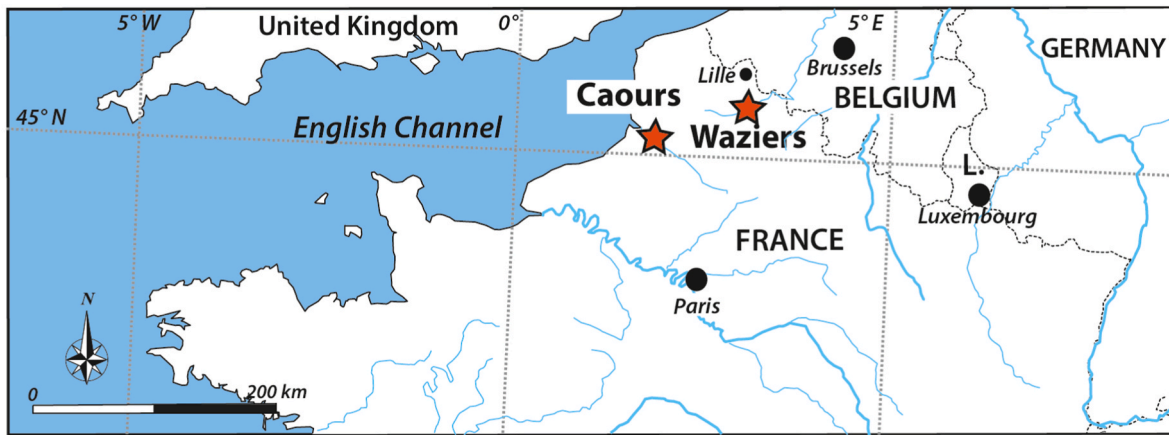


Fig. 1. Location of the studied sites in northern France.

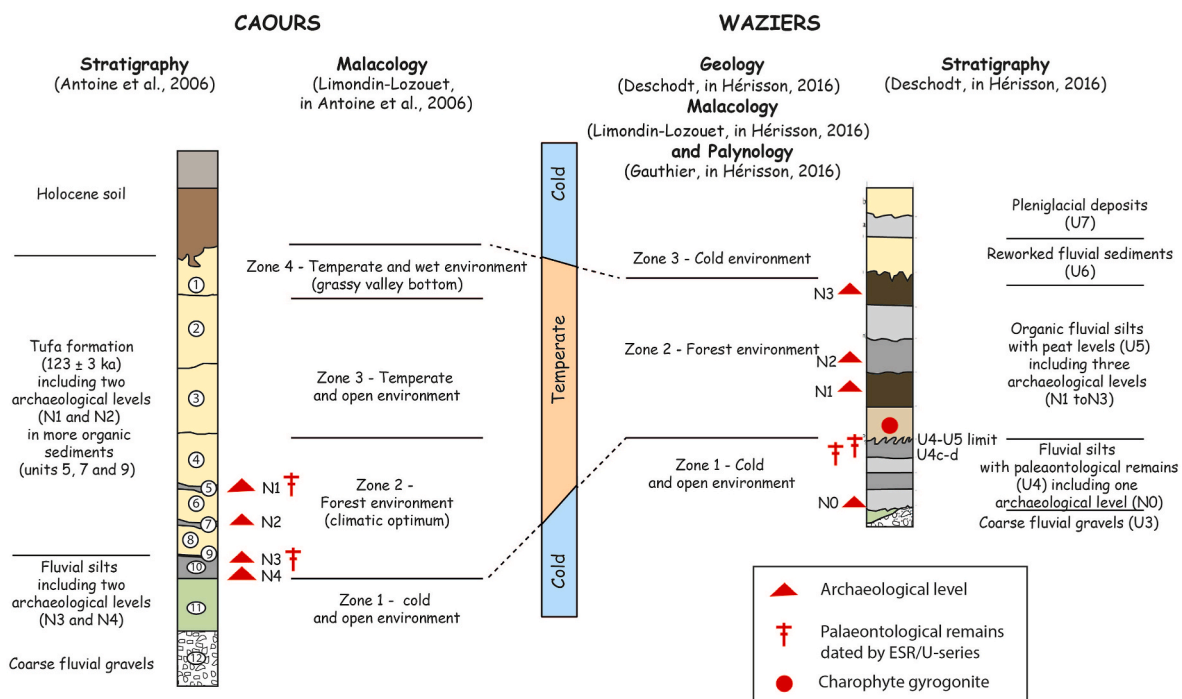


Fig. 2. Summary of stratigraphic and palaeoenvironmental data available from the two studied Eemian sites from northern France (Caours and Waziers). The analysed teeth were recovered from archaeological levels N1 and N3 at Caours and from units U4c-d and U4–U5 limit at Waziers.

European chronostratigraphy (Cohen and Gibbard, 2019).

Palaeolithic sites associated with the Eemian interglacial (MIS 5e) are very rare in northern France Quaternary sequences owing to the major erosion processes occurring during the early stages of the subsequent Weichselian period (Antoine et al., 2016). In the 1980s some researchers considered even a lack of human presence in North-Western Europe during this interglacial (Gamble, 1896).

In northern France, only two Eemian sites have been discovered so far and are currently excavated: Caours (Somme) and Waziers (Nord) (Fig. 1) (Locht et al., 2014, 2016; Hérisson, 2016).

## 2. Site presentation and sampling

As mentioned previously, Eemian archaeological sites are quite rare in northern France, which probably does not reflect the absence of human groups in this area at this time but rather taphonomic issues (Roebroeks and Speleers, 2002). As explained by Antoine et al. (2002,

2016) and Loch et al. (2014, 2016), the main part of these sites has probably been destroyed or displaced during the earliest cold events of the Weichselian, beginning ca 116 ka ago (Sakari Salonen et al., 2018). Until the beginning of the 2000s, the Eemian archaeological evidences in northern France loess-palaeosol sequences were hence restricted to materials reworked by erosion (Antoine et al., 2002). Known since the XIX<sup>th</sup> century (Prestwich et al., 1864), the Caours site was rediscovered in 2002 (Antoine et al., 2006), in the frame of a research program focusing on interglacial deposits in the Somme terraces system (Antoine et al., 2007). The subsequent discovery of the Waziers locality in 2011 during a campaign of rescue survey, definitively demonstrated that human groups were present in northern France during the Eemian (Locht et al., 2014, 2016; Hérisson, 2016).

The Caours site, located close to Abbeville in the Scardon Valley, a small tributary of the Somme River, was known since the 1940s (Auffrère, cited in Breuil, 1952; Bourdier, 1969), but never really studied and published at that time. The new investigation since 2002 shows that

the site is characterized by a thick calcareous tufa formation (3–4 m) overlying periglacial fluvial flint gravels attributed to the penultimate glacial stage (Saalian, MIS 6) (Fig. 2) (Antoine et al., 2006, 2007; Dabkowski et al., 2015, 2016). The lower part of the Caours stratigraphical sequence fossilized four archaeological levels (Antoine et al., 2006; Locht et al., 2016) containing abundant Middle Palaeolithic artefacts. The associated interglacial faunal remains correspond to a well-developed forested temperate environment where some open area spaces as meadows persisted (Auguste, 2009). Uranium-series (U-series) on carbonates, thermoluminescence (TL) on heated flints and optically stimulated luminescence (OSL) on sediments dates constrain the tufa formation and Caours archaeological levels to  $123 \pm 3$  ka, allowing their attribution to the Eemian (Antoine et al., 2006, 2007; Antoine and Locht, 2015). The malacofauna study permits to precise the position of the Eemian climatic optimum in the lower part of the tufa formation (units 4–10), the lowest part of the sequence being assigned to the late Saalian glacial (unit 11) and the top of the tufa (units 1–3) related to a climate drying during the subsequent interglacial stage (Limondin-Lozouet, in Antoine et al., 2006; Limondin-Lozouet, 2011; Limondin-Lozouet and Preece, 2014). Five teeth from the interglacial part of the sequence, four from archaeological level N1 (unit 5) and one from N3 (unit 9), were sampled and analysed in the present work by combined electron spin resonance and U-series dating method (ESR/U-series). Another tooth from N1 level was previously dated by the same method to  $124 \pm 15$  ka (Bahain et al., 2010).

Waziers is located close to Douai, in the Scarpe river valley. The site was discovered during archaeological survey operation in 2011 and 2013, a first sector being excavated in 2014–2015 (Hérissou, 2016). The stratigraphic sequence (Fig. 2) encloses coarse fluvial deposits (sands and gravels) recovered by calcareous silts, organic silts and peat levels infilling a large palaeo-meander channel of the Scarpe River and then covered by chalky gravels and finally by sandy loess deposits (Deschodt, in Hérissou, 2016). The organic silts and peat layers yielded four archaeological levels (N0–N3) with some Middle Palaeolithic flint artefacts associated with large mammal remains including well preserved mammal teeth. U-series minimum age ( $103 + 3,5/-3,4$  ka) on charophyte gyrogonite extracted from calcareous sandy silt at the very base of the interglacial sequence infilling the channel (Ghaleb, in Hérissou, 2016) and the malacological study (Limondin-Lozouet, in Hérissou, 2016) indicate the Pleistocene age of the sequence. Malacological and mainly palynological data (Gauthier, in Hérissou, 2016) allow the description of a vegetation dynamic corresponding to the transition between a late glacial stage and an interglacial stage, then to an interglacial record. Indeed they demonstrate both open landscape and cold environment in the fluvial deposits of the bottom of the sequence (units 3 and 4). Then, temperate environment conditions are recorded through

the successive development of pioneer trees then of deciduous forest phases (*Ulmus*, *Quercus*, *Corylus*, lastly *Carpinus* as dominant trees) corresponding to an interglacial while locally the site progress from a peat bog to a swamp forest. The mammal fauna associated to these interglacial levels is, as at Caours, typical of a forested environment, while species characteristics of more open area as equids were recovered in the lower silty fluvial deposits and the position of the site in the valley as well as the presence of Middle Palaeolithic industry in several levels has allowed the attribution of the stratigraphic sequence to the late glacial phase of the Saalian glacial (MIS 6) and to the Eemian interglacial (Auguste, in Hérissou, 2016). In the present work, four teeth from Waziers, coming from the late glacial fluvial deposits, were analysed, three from unit 4c-d and one at the boundary between units 4 and 5 (Fig. 2).

### 3. Methods and protocols

ESR/U-series dating method was applied using the protocol of preparation and analyses described in Bahain et al. (2010). Enamel layers were mechanically extracted from the teeth and cleaned using a dentist drill to remove dentine or sediment contamination. The clean enamel sample was then ground and the 100–200  $\mu\text{m}$  grain-size fraction was split into 10 (Caours) or 14 (Waziers) aliquots. One aliquot was kept as “natural” reference and the other aliquots were gamma irradiated at doses ranging from 50 to 2,000 Gy (Caours, IBL  $^{60}\text{Co}$  source, CEN Saclay, France) or from 25 to 6,300 Gy (Waziers, Gammacell  $^{137}\text{Cs}$  source, CENIEH, Burgos, Spain) with a dose rate of about 200 Gy/h.

The ESR intensity of each aliquot was then measured using a Bruker EMX spectrometer at room temperature using X band (frequency modulation of 100 kHz) at MNHN, Paris, France. The following parameters were used for the measurements: microwave power of 10 mW, modulation amplitude of 0.1 mT, scan range of 10 mT, scan time of 4 min. At least four measurements were performed for each aliquot on different days. The ESR intensities of the radiation-induced of the enamel signal at  $g = 2.00018$  were measured peak-to-peak (T1-B2) according to Grün (2000). Equivalent doses were determined from the obtained dose-intensity data points using single exponential function with  $1/I^2$  weighting.

U-series analyses were performed on each dental tissue, by alpha spectrometry (Caours samples) at MNHN, Paris, after chemical preparation preconized by Bischoff et al. (1988), or by Multi-Collector Inductively Coupled Plasma Mass Spectrometer (Neptune MC-ICPMS) (Waziers samples) at Nanjing Normal University, China with the chemical protocol of Shao et al. (2015a). Eventual radon loss was determined from each tissue by combining alpha-ray and gamma-ray data (Bahain et al., 1992). U-series data are displayed Table 1.

**Table 1**

U-series data obtained on the dental tissues of the analysed teeth from two Eemian sites of Northern France (Caours and Waziers). The ages are calculated with  $1 \sigma$  error range.

Sample	Level	Tissue	U (ppm)	$^{230}\text{Th}/^{232}\text{Th}$	$^{234}\text{U}/^{238}\text{U}$	$^{230}\text{Th}/^{234}\text{U}$	$^{222}\text{Rn}/^{230}\text{Th}$	Apparent U-series age (ka)
CA-0501	US 5 - N1	Enamel	$0.332 \pm 0.020$	12	$1.376 \pm 0.094$	$0.927 \pm 0.086$	1.00	$220 \pm 97$
		Dentine	$8.235 \pm 0.220$	318	$1.281 \pm 0.032$	$1.342 \pm 0.052$	0.17	>300
CA-0502	US 5 - N1	Enamel	$0.301 \pm 0.017$	16	$1.247 \pm 0.075$	$0.813 \pm 0.078$	0.55	$166 \pm 48$
		Dentine	$6.560 \pm 0.219$	90	$1.232 \pm 0.044$	$1.419 \pm 0.080$	0.32	>300
CA-0503	US 5 - N1	Enamel	$0.393 \pm 0.029$	23	$1.063 \pm 0.094$	$0.953 \pm 0.102$	1.00	$299 \pm 150$
		Dentine	$10.270 \pm 0.242$	133	$1.167 \pm 0.026$	$1.150 \pm 0.042$	0.16	>300
CA-0701	US 5 - N1	Enamel	$0.338 \pm 0.016$	8	$1.274 \pm 0.066$	$0.923 \pm 0.055$	0.92	$226 \pm 62$
		Dentine	$5.281 \pm 0.165$	113	$1.229 \pm 0.014$	$1.008 \pm 0.052$	0.14	$327 \pm 172$
CA-0504	US 9 - N3	Enamel	$0.193 \pm 0.012$	13	$1.222 \pm 0.090$	$0.884 \pm 0.087$	1.00	$205 \pm 96$
		Dentine	$13.771 \pm 0.316$	67	$1.225 \pm 0.021$	$1.135 \pm 0.048$	0.09	>300
WBT-138	limit US4-US5	Enamel	$0.070 \pm 0.001$	344	$1.175 \pm 0.002$	$0.687 \pm 0.003$	1.00	$121 \pm 1$
		Dentine	$0.068 \pm 0.001$	13	$1.354 \pm 0.006$	$0.679 \pm 0.004$	1.00	$111 \pm 3$
WBT-19	US 4c-d	Enamel	$0.036 \pm 0.001$	46	$1.467 \pm 0.017$	$0.600 \pm 0.009$	1.00	$93 \pm 2$
		Dentine	$0.097 \pm 0.001$	128	$1.609 \pm 0.004$	$0.547 \pm 0.002$	1.00	$81 \pm 1$
WBT-22-1	US 4c-d	Enamel	$0.071 \pm 0.001$	213	$1.246 \pm 0.005$	$0.775 \pm 0.004$	1.00	$150 \pm 2$
		Dentine	$0.028 \pm 0.001$	16	$1.348 \pm 0.005$	$0.655 \pm 0.004$	1.00	$106 \pm 2$
WBT-22-2	US 4c-d	Enamel	$0.009 \pm 0.001$	30	$1.386 \pm 0.011$	$0.672 \pm 0.008$	1.00	$111 \pm 3$
		Dentine	$0.015 \pm 0.001$	145	$1.508 \pm 0.007$	$0.555 \pm 0.0043$	1.00	$83 \pm 1$

**Table 2**

Radioelements contents of sediments associated to the analysed teeth from two Eemian sites of Northern France (Caours and Waziers).

Sample	Level	<sup>238</sup> U (ppm)	<sup>232</sup> Th (ppm)	<sup>4</sup> K (%)	Water content (%)
CA-0501	US 5 - N1	0.627 ± 0.093	1.002 ± 0.164	0.202 ± 0.011	15 ± 5
CA-0502	US 5 - N1	1.072 ± 0.123	3.887 ± 0.210	0.859 ± 0.019	15 ± 5
CA-0503	US 5 - N1	0.383 ± 0.071	0.130 ± 0.005	0.039 ± 0.005	15 ± 5
CA-0701	US 5 - N1	0.286 ± 0.046	0.671 ± 0.053	0.116 ± 0.005	15 ± 5
CA-0504	US 9 - N3	1.219 ± 0.102	6.242 ± 0.183	1.169 ± 0.016	15 ± 5
WBT-138	Limit US 4-5	0.379 ± 0.085	3.249 ± 0.115	0.957 ± 0.016	28 ± 5
WBT-19, 22-1 & 22-2	US 4c-d	0.558 ± 0.077	3.340 ± 0.107	0.942 ± 0.014	20 ± 5

The dose rate was stated from measurements by gamma-ray spectrometry in laboratory from 100 g of sediments corresponding to the analysed teeth with a low background high purity germanium (HPGe) detector (Table 2). No disequilibrium in the natural radioactive chains was observed in the sediments of the two sites. Water content of sediments was previously measured for each dated level by drying it in oven at 40 °C during one week. The cosmic dose rate was estimated using the formula of Prescott and Hutton (1994).

*In situ* measurements were also performed using Canberra Inspector1000 spectrometer (Mercier and Falguères, 2007): at Caours, which is a yearly excavated site, these measurements were made in the immediate vicinity of each analysed sample and several measurements were hence performed for the same N1 archaeological level; at Waziers, as the site is close to a road and the sections not conserved for a long time, the *in situ* dosimetry was performed on the single available section, explaining why only one measurement was performed per dated layer. At Caours, the *in situ* measurements are globally lower than the dose rates calculated from the radioelement contents of the sediments while at Waziers the data are quite similar for the two kinds of measurements.

The U-uptake parameters, dose rate contributions and ESR/U-series ages with their uncertainties were then calculated (see details in Shao et al., 2012, 2014; 2015b). As the U-series results on several dental tissues were older than the corresponding EU-ESR data, it was not possible to systematically apply the US model (Grün et al., 1988) and the accelerating U-uptake (AU) model by Shao et al. (2012), allowing the modelling of U-leaching from some of the tissues, was then used instead. The age calculations were performed using the “AUAGE” and “USESR” computer programs with the following parameters: a k-value ( $\alpha$  efficiency) of  $0.13 \pm 0.02$  (Grün and Katzenberger-Apel, 1994); water contents of 0 wt% in the enamel and 7 wt% in the dentine; radioelements content-dose conversion factors of Guérin et al. (2011); beta attenuations values in dental tissues were calculated using a Monte Carlo approach taking into account the enamel thickness removed during the sample preparation (Brennan et al., 1997) (Table 3).

**Table 3**

Initial and mechanically removed thicknesses of enamel layers during the preparation of the analysed teeth from two Eemian sites of Northern France (Caours and Waziers) prior to ESR/U-series dating.

Sample	Level	Initial thickness ( $\mu\text{m}$ )	Removed thickness	
			Internal side ( $\mu\text{m}$ )	External side ( $\mu\text{m}$ )
CA-0501	US 5 - N1	867 ± 108	27 ± 3	4 ± 1
CA-0502	US 5 - N1	993 ± 124	50 ± 6	20 ± 2
CA-0503	US 5 - N1	893 ± 112	79 ± 7	4 ± 1
CA-0701	US 5 - N1	957 ± 120	43 ± 5	17 ± 2
CA-0504	US 9 - N3	931 ± 230	169 ± 18	103 ± 25
WBT-138	Limit US 4-5	1250 ± 156	38 ± 5	135 ± 17
WBT-19	US 4c-d	1209 ± 151	70 ± 9	84 ± 11
WBT-22-1	US 4c-d	1113 ± 139	55 ± 7	39 ± 5
WBT-22-2	US 4c-d	883 ± 110	39 ± 5	75 ± 9

#### 4. Results and discussion

The ESR/U-series results obtained on the teeth from the two Eemian sites of northern France are shown in Table 4, while Fig. 3 displays the dose rate contributions (both in  $\mu\text{Gy/a}$  and in %) for the different analysed teeth.

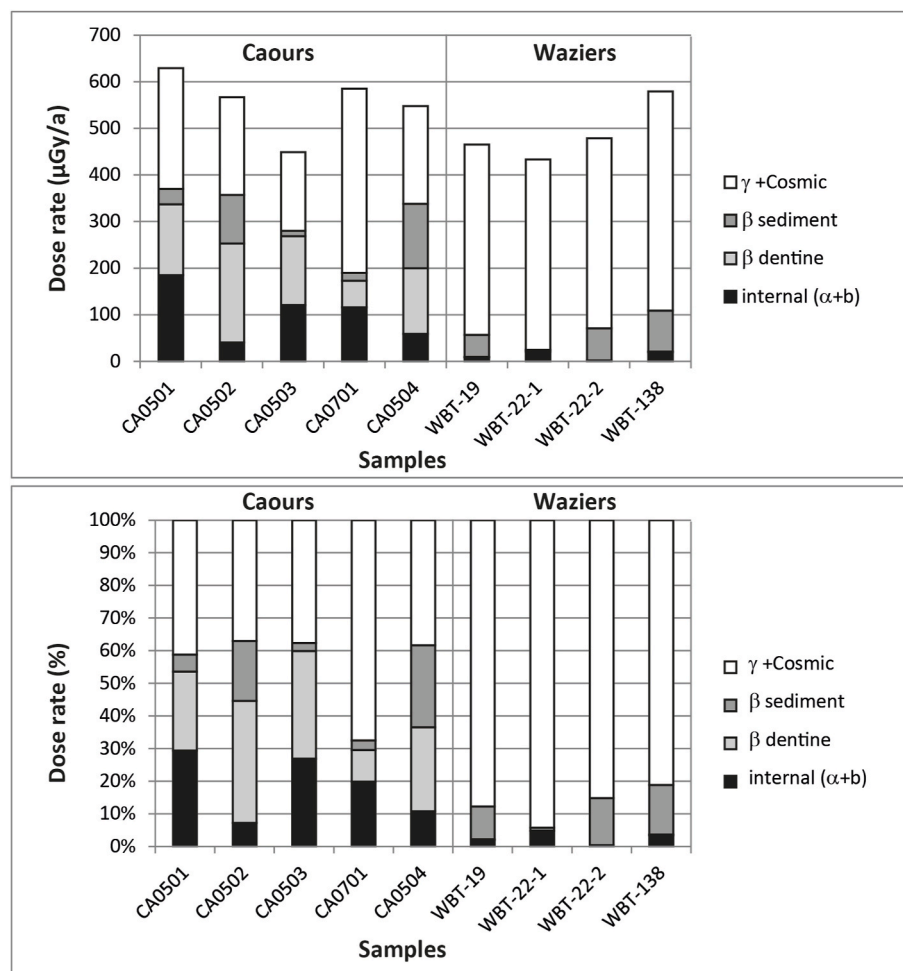
Several interesting points can be highlighted in these data:

- The Waziers dental tissues display very low uranium contents,  $< 0.1$  ppm in both enamel and dentine of all teeth. Moreover, the enamel often shows higher concentrations than in the corresponding dentine, which is very unusual. This behavior is perhaps linked to the reducing environment associated with the decomposition of the organic plant matter in the peat, leading to the fixation of the uranium in the humic acids of the peat rather than in the paleontological remains, whereas thorium will be absorbed on the detrital particles (Geyh, 2008; Waas et al., 2011);
- In consequence of this very specific geochemical behavior, the internal dose has a restricted impact in the age determination as well as the  $\beta$  dose contribution related to the dentines ( $< 20\%$ ) compared to the external (sediment + cosmic) dose (Fig. 3).
- By comparison, U contents of the Caours dental tissues range from  $\sim 0.19$  to  $\sim 0.39$  ppm in the enamels and from  $\sim 5.28$  to  $\sim 13.77$  ppm in the dentines. They correspond to more usual values and leads to a greater contribution of the Caours dental tissues to the dose rate, ranging between 30 and 60%.
- U-series analyses reveal on the other hand high  $^{230}\text{Th}/^{234}\text{U}$  ratios (between 0.813 and 1.342) in the tissues of Caours teeth, leading to apparent U-series ages higher than the EU-ESR age, which indicate a possible U-leaching.
- As a consequence, only the AU model was used to calculate ESR/U-series ages for the Caours samples, this model considering an initial U-uptake followed by a U-leaching. This U-leaching could be related to the growth of the overlying tufa formation, implying phases of important water circulation. This leaching, combined with the significant errors on the U-series isotopic ratios determined by  $\alpha$ -spectrometry U-series analyses on the Caours dental tissues, leads to large age uncertainties for some teeth (e.g. CA-0501 and CA-0502).
- For the Waziers tissues, the apparent U-series ages are close to the EU-ESR ages (120–130 ka) except for the enamel of tooth WBT-22-1 for which the obtained value is higher. As previously mentioned, this modelling has however not impact on the age calculation due to the poor associated dose contribution.
- On each site, the equivalent doses are quite low (Fig. 4). For the Caours samples, the  $D_E$  range between 50 and 80 Gy, but these variations are counterbalanced by the local variation of external doses, precisely recorded at the sampling location on the site for each tooth. This work demonstrates hence the importance of an accurate *in situ* dosimetry in the age calculation for such low radioactive environment depending of the repartition of the palaeontological remains that can represent radioactivity hotspots. The ESR/U-series ages for Caours teeth determined using AU model for N1 teeth are relatively homogeneous, ranging from  $119 \pm 42$  ka to  $126 \pm 37$  ka and leading to a mean age of  $124 \pm 10$  ka ( $\pm 2\sigma$ ) for level N1

**Table 4**

Equivalent doses, U-uptake parameters, contributions to the dose rate and ESR/U-series ages obtained for the analysed teeth from two Eemian sites of Northern France (Caours and Waziers). The ESR-U-series ages are given with  $\pm 2$  sigma.

Sample	Level	Tissue	Equivalent dose $D_E$ (Gy)	U uptake parameters $p$ (regular) or $n$ ( <i>italics</i> )	$D_a \alpha$ internal ( $\mu\text{Gy/a}$ )	$D_a \beta$ ( $\mu\text{Gy/a}$ )	$D_a (\gamma + \text{cosm})$ ( $\mu\text{Gy/a}$ )	$D_a$ total ( $\mu\text{Gy/a}$ )	ESR/U-series ages US (regular) or AU ( <i>italics</i> ) models (ka)
CA-0501	N1- U5	Enamel	79.31 $\pm$ 3.07	-0.0123 $\pm$ 0.0037	185 $\pm$ 143	185 $\pm$ 117	259 $\pm$ 21	629 $\pm$ 186	126 $\pm$ 37
		Dentine		-0.0139 $\pm$ 0.0098					
CA-0502	N1- U5	Enamel	67.53 $\pm$ 2.66	-0.0128 $\pm$ 0.0051	41 $\pm$ 37	316 $\pm$ 196	210 $\pm$ 20	567 $\pm$ 201	119 $\pm$ 42
		Dentine		+0.0390 $\pm$ 0.1431					
CA-0503	N1- U5	Enamel	53.39 $\pm$ 5.30	-0.0088 $\pm$ 0.0005	121 $\pm$ 3	159 $\pm$ 10	169 $\pm$ 22	449 $\pm$ 24	119 $\pm$ 15
		Dentine		-0.0105 $\pm$ 0.0005					
CA-0701	N1- U5	Enamel	73.16 $\pm$ 2.90	-0.0077 $\pm$ 0.0001	116 $\pm$ 1	74 $\pm$ 3	395 $\pm$ 20	585 $\pm$ 20	125 $\pm$ 5
		Dentine		-0.0104 $\pm$ 0.0002					
CA-0504	N3-U9	Enamel	71.00 $\pm$ 10.62	-0.0312 $\pm$ 0.0057	59 $\pm$ 2	279 $\pm$ 21	210 $\pm$ 20	548 $\pm$ 29	129 $\pm$ 23
		Dentine		-0.0102 $\pm$ 0.0008					
WBT-138	Limit US 4-5	Email	76.47 $\pm$ 2.72	-0.9386 $\pm$ 0.0461	20 $\pm$ 32	89 $\pm$ 12	470 $\pm$ 23	579 $\pm$ 41	132 $\pm$ 8
		Dentine		-0.9016 $\pm$ 0.0479					
WBT-19	US4c-d	Email	58.08 $\pm$ 1.70	-0.7551 $\pm$ 0.0608	9 $\pm$ 22	48 $\pm$ 9	408 $\pm$ 20	465 $\pm$ 33	125 $\pm$ 8
		Dentine		-0.5956 $\pm$ 0.0711					
WBT-22-1	US4c-d	Email	59.71 $\pm$ 1.03	-0.0106 $\pm$ 0.0010	21 $\pm$ 3	4 $\pm$ 42	408 $\pm$ 20	433 $\pm$ 26	138 $\pm$ 8
		Dentine		-0.8125 $\pm$ 0.0502					
WBT-22-2	US4c-d	Email	58.95 $\pm$ 1.66	-0.9384 $\pm$ 0.0438	2 $\pm$ 20	69 $\pm$ 10	408 $\pm$ 20	479 $\pm$ 30	123 $\pm$ 7
		Dentine		-0.6443 $\pm$ 0.0608					



**Fig. 3.** Dose rate contributions calculated for the analysed teeth of two Eemian sites of northern France (Caours and Waziers).

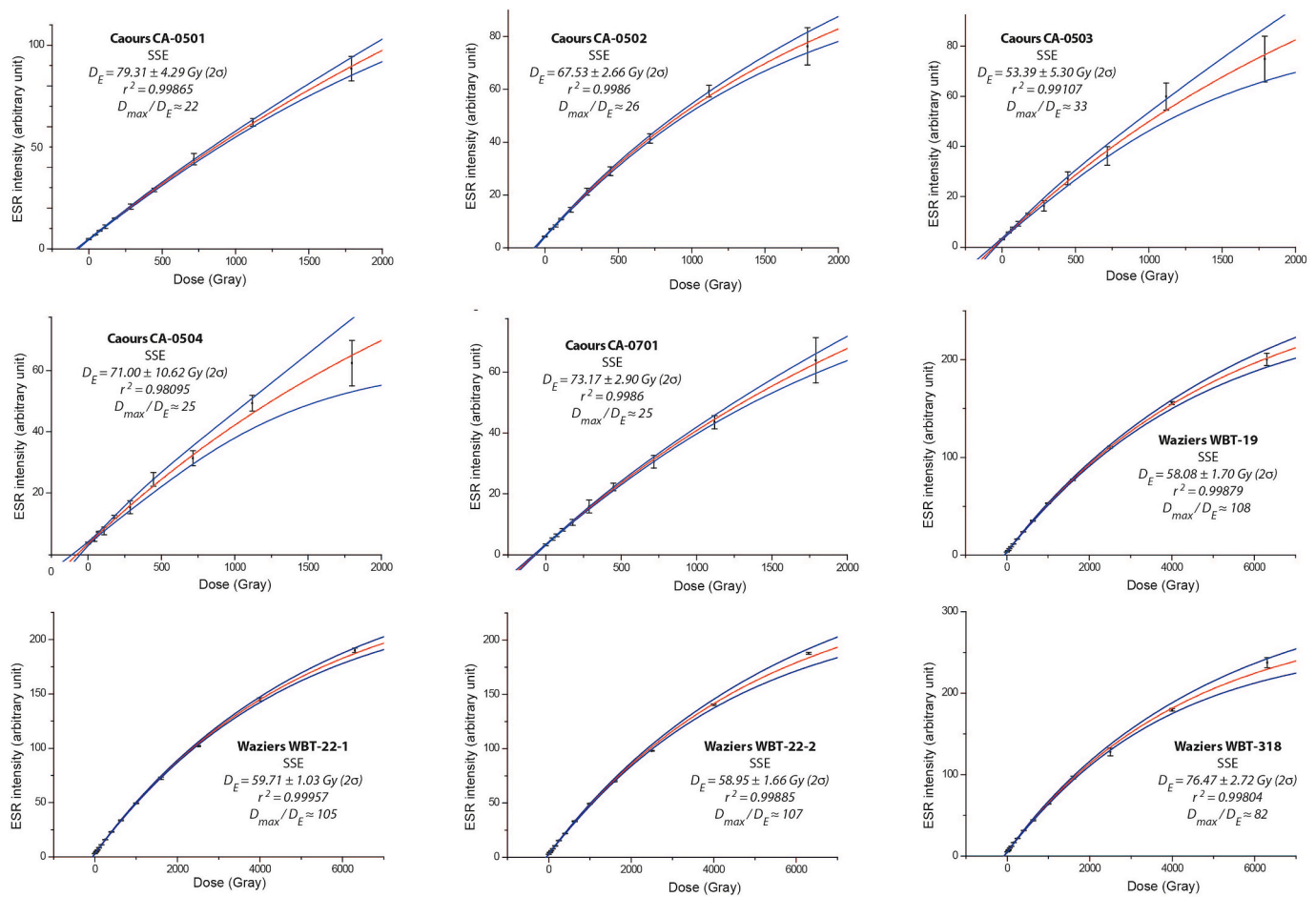


Fig. 4. Dose response curves obtained for the analysed teeth of two Eemian sites of northern France (Caours and Waziers).

(calculated with Isoplot, Ludwig, 2003), similar to the age of  $124 \pm 15$  ka obtained previously by Bahain et al. (2010). The tooth analysed from level N3 provides an AU age of  $129 \pm 23$  ka. Given these very homogeneous ages for the two dated levels, the attribution of the deposit to the Eemian interglacial is confirmed with an ESR/U-series mean age of  $125 \pm 9$  ka.

- At Waziers, despite the impossibility to measure so precisely the external dose rate variations due to a more restricted access to the dated level that could explain the scattering of the age results obtained for the limit US 4–5 teeth, the homogeneity of the equivalent doses obtained for the teeth of this level, comprised between 58 and 60 Gy, leads to the calculation of a weighted mean age of  $128 \pm 20$  ka ( $\pm 2\sigma$ ), while the higher  $D_E$  value obtained for the U4c-d level (76 Gy) probably in relation with a more radioactive background, leads to an undistinguishable US age of  $132 \pm 8$  ka. Given these very homogeneous ages for the two dated levels, a weighted mean age of  $129 \pm 11$  ka ( $2\sigma$ ) can be calculated from these four samples. This is in good agreement with the attribution of the deposition of the fluvial silts of Waziers U4 level to the Saalian late glacial and consistent with the available palaeoenvironmental data.

### 5. Conclusion

The combined ESR/U-series analyses performed on fossil teeth

collected from two Eemian sites of northern France, Caours and Waziers, demonstrate the reliability of the method to date such Middle Paleolithic sites (Fig. 5). The results also indirectly emphasize the validity of the analytical procedures employed, despite peculiar geochemical characteristics of part of the dental tissues. Finally, this work also shows in particular the importance of the *in situ* dosimetry measurements in the Caours age calculation.

At Caours, the obtained ESR/U-series ages lead to the calculation of a weighted mean age of  $125 \pm 9$  ka in excellent agreement with the other available geochronological data derived from U-series and luminescence studies and dating the overlying tufa formation of  $123 \pm 3$  ka. Associated to the available palaeoenvironmental, mammal's association and archaeological data, they make of this site one of the best dated Middle Palaeolithic sequence of France.

For Waziers, the newly established ESR/U-series chronology for teeth of Unit 4, with a mean age of  $129 \pm 11$  ka, represents the first numerical age estimate for these levels. It confirms its attribution to the Saalian late glacial stage (MIS6), one more time in close agreement with the palaeoenvironmental data, and allows the attribution to the overlying organic fluvial deposits and associated archaeological levels to the Eemian interglacial.

Lastly, these results show the interest to integrate ESR/U-series dating analyses during the chronostratigraphical study of such Middle Palaeolithic sites when possible and demonstrate the necessity to

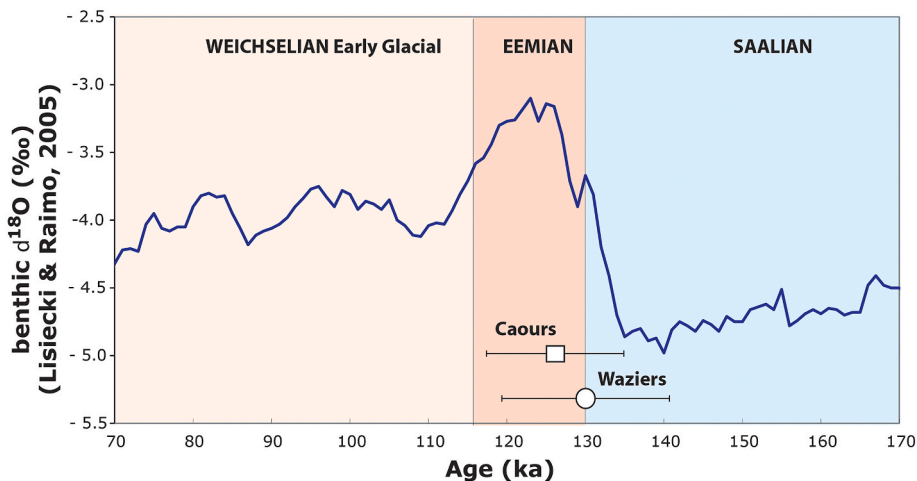


Fig. 5. Geochronological correlation of the obtained ESR/U-series mean ages for two Eemian sites of northern France (Caours and Waziers) with the Marine isotopic record of Lisiecki and Raymo (2005).

involve geochronologists to the fieldworks and excavations, since the earliest stages of study.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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