

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

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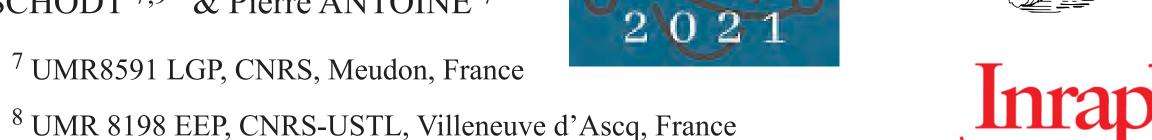
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#### Introduction

Palaeolithic sites associated with the Eemian Interglacial (MIS 5e) are very rare in Northern France and their preservation is restricted to very specific geological contexts, in association with carbonated tufa (Caours) or peat deposits (Waziers) (Figure 1).

ESR/U-series dating method was hence applied on teeth recovered from different levels of the two sites, in the aim to evaluate the quality of the results obtained in very clearly chronologically constrained stratigraphic sequences. The results obtained will be displayed and discussed in this





# Material and method

ESR/U-series dating method was applied using the protocol of preparation and analyses described in Bahain et al. (2010).

Equivalent doses were determined using single exponential function. The dose rate was estimated from measurements by gamma-ray spectrometry, both in laboratory with a low background, high purity germanium detector and in situ using Canberra Inspector 1000 spectrometer. Rn loss from each tissue was determined by combining alpha-ray and gamma-ray data (Bahain et al., 1992). The cosmic dose rate was estimated using the formula of Prescott and Hutton (1994).

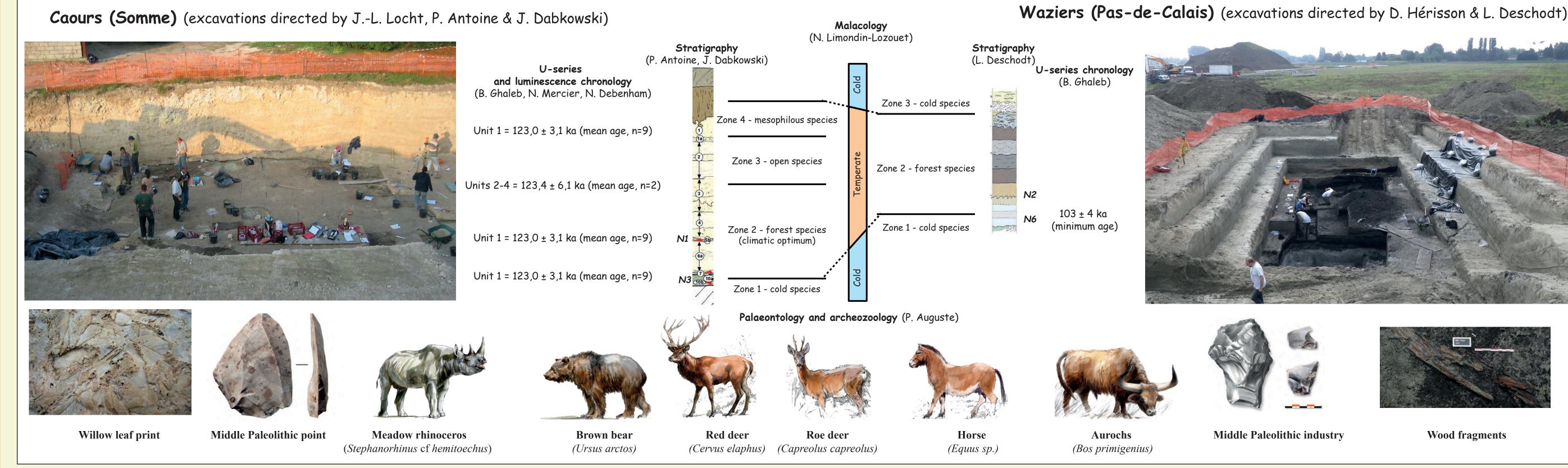
The following parameters were also used in the age determination:

- a k-value of  $0.13 \pm 0.02$  (Grün & Katzenberger-Apel, 1994);
- water content of sediments was measured for each dated level by drying it in oven at 40°C during one week;
- water contents of 0 wt% in the enamel and 7 wt% in the dentine;
- conversion factors of Adamiec and Aitken (1998);
- beta attenuations values in dental tissues were calculated using a Monte Carlo approach (Brennan et al., 1997).

#### Site presentation and sampling

Since 2003 for Caours and 2011 for Wasiers, these two sites are studied by multidisciplinary teams including geologists, archaeologists, paleontologists, malacologists and geochronologists. The main results of these studies are summarized in Figure 2.

**Figure 2** - Presentation of the Caours and Waziers Eemian sites in Northern France.



The Caours site is located close to Abbeville in the Scardon Valley. Coarse fluvial deposits were here recovered by silts and a mm thick tufa sequence who fossilized several archeological levels containing abundant Middle Paleolithic industry and faunal remains associated with a forested temperate environment. U-series, TL and OSL dates constrain the tufa formation and Caours archeological level to ca 123 ka, allowing its clear attribution to Eemian interglacial stage (MIS 5e). Malacofauna study permits to place the climatic optimum of this stage in the lower part of the tufa formation.

Five teeth, four from archaeological level N1 and one from N3, were analyzed in the present work by ESR/U-series. Another tooth was previously dated by the same method of  $124 \pm 15$  ka (Bahain et al., 2010).

Waziers site is located close to Douai, in the Scarpe river valley. Discovered during archeological survey operation in 2011 et 2013, it was excavated in 2014-2015. The stratigraphic sequence encloses coarse fluvial deposits recovered by an alternation of silts and peat levels including archaeological levels with Middle Palaeolithic lithic industries, then by sandy loess deposits. Malacofauna study indicates at the bottom of the sequence both open landscape and cold environement, then an interglacial temperate environment with first peat bog then swampy forest. The associated mammal fauna is typical from forested environment also (cervids, aurochs, beavers,...). Four teeth from Waziers were analyzed during the present work, three from N2 and one from N6.

# Results

The ESR/U-series results are shown in Table 1. Several interesting points can be highlighted in these data.

#### Table 1. ESR/U-series results obtained on teeth recovered from two Eemian sites of Northern France

Site	Unit	Sample	Tissue	U (ppm)	234U/238U	<sup>230</sup> Th/ <sup>232</sup> Th	<sup>230</sup> Th/ <sup>234</sup> U	<sup>222</sup> Rn/ <sup>230</sup> Th	Initial thickness (μm)	Rem. thickness internal side (μm)	Rem. thickness external side (µm)	S Equivalent doses (Gy)	U-uptake parameter (p or n)	Internal D <sub>a</sub> (μGy/an)	D <sub>a</sub> β (μGy/an)	D <sub>a</sub> (γ+cosm) (μGy/an)	D <sub>a</sub> (μGy/an)	ESR/U-series ages (US or AU) (ka)
	<b>N</b> T1	C 4 2005 01	dentine	$8.235 \pm 0.220$	$1.281 \pm 0.032$	318	$1.342 \pm 0.052$	0.17	0.67 + 100	27 + 2	4 + 1	70.21 + 2.07	-0.0139 ± 0.0098	105 : 142	105 - 117	250 + 21	(20 + 10)	124 + 27
	N1	CA2005-01	enamel	$0.332 \pm 0.020$	$1.376 \pm 0.094$	12	$\underline{0.927 \pm 0.086}$	1.00	$867 \pm 108$	27 ± 3	4 ± 1	$79.31 \pm 3.07$	0.0123 ± 0.0037	185 ± 143	185 ± 117	259 ± 21	629 ± 186	126 ± 37
Caours	N1	CA2005-02	dentine	$6.560 \pm 0.219$	$1.232 \pm 0.044$	90	$1.419 \pm 0.080$	0.32	993 ± 124	50 ± 6	20 ± 2	65.50 . 0.66	$+0.0390 \pm 0.1431$	41 + 27	216 + 106	210 + 20	567 + 201	110 + 42
			enamel	$0.301 \pm 0.017$	$1.247 \pm 0.075$	16	$0.813 \pm 0.078$	0.55					-0.0128 ± 0.0051	41 ± 37 	316 ± 196 	210 ± 20	567 ± 201	119 ± 42
	NT1	CA2005-03	dentine	$10.270 \pm 0.242$	$1.167 \pm 0.026$	133	$1.150 \pm 0.042$	0.16	893 ± 119	59 ± 7	4 ± 1	53.39 ± 5.30	$-0.0105 \pm 0.0005$	121 ± 3	159 ± 10	169 ± 22	449 ± 24	119 ± 15
	N1		enamel	$0.393 \pm 0.029$	$1.063 \pm 0.094$	23	$0.953 \pm 0.102$	1.00					-0.0088 ± 0.0005					
	NIO	CA2005-04	dentine	$13.771 \pm 0.316$	$1.225 \pm 0.021$	67	$1.135 \pm 0.048$	0.09	1014 ± 127	43 ± 5	17 ± 2	73.16 ± 2.90	$-0.0104 \pm 0.0002$	116 ± 1	74 ± 3	395 ± 20	585 ± 20	125 ± 5
	N3		enamel	$0.193 \pm 0.012$	$1.222 \pm 0.090$	13	$0.884 \pm 0.087$	1.00										
	NI1	CA 2007 01	dentine	$5.281 \pm 0.165$	$1.229 \pm 0.014$	113	$1.008 \pm 0.052$	0.14	057 + 120	$2\pm0$	$29 \pm 4$		$-0.0102 \pm 0.0008$		279 ± 21	$210 \pm 20$	548 ± 29	$129 \pm 23$
	N1	CA2007-01	enamel	$0.338 \pm 0.016$	$1.274 \pm 0.066$	8	$0.923 \pm 0.055$	0.92	$957 \pm 120$	2 ± 0	29 ± 4	$71.00 \pm 10.02$	-0.0312 ± 0.0057	J9 ± 2	$2/9 \pm 21$	$210 \pm 20$	J40 ± 29	129 ± 23
Waziers	2	WBT-19	dentine	$0.097 \pm 0.001$	$1.609 \pm 0.004$	128	$0.547 \pm 0.003$	1.00	$1250 \pm 156$	38 ± 5	135 ± 17	58.08 ± 1.70	$-0.0127 \pm 0.0017$	9 ± 22	48 ± 9	408 ± 20	465 ± 33	125 ± 8
			enamel	$0.036 \pm 0.001$	$1.467 \pm 0.017$	46	$0.600 \pm 0.009$	1.00					$-0.7551 \pm 0.0608$					
	2	WBT-22-1	dentine	$0.028 \pm 0.001$	$1.348 \pm 0.005$	 16	$0.655 \pm 0.004$	1.00	$1209 \pm 151$	70 ± 9	84 ± 11	$59.71 \pm 1.03$	$-0.8125 \pm 0.0502$					
			enamel	$0.071 \pm 0.001$	$1.246 \pm 0.005$ .	213	$0.775 \pm 0.004$	1.00					-0.0106 ± 0.0010	$21 \pm 3$	$4 \pm 42$	$408 \pm 20$	$433 \pm 26$	$138 \pm 8$
	2	WBT-22-2	dentine	$0.015 \pm 0.001$	- $        -$	 145	$       0.555 \pm 0.004$	1.00	$1113 \pm 139$	55 ± 7	39 ± 5	$58.95 \pm 1.66$	$-0.6443 \pm 0.0608$		69 ± 10	408 ± 20	479 ± 30	123 ± 7
			enamel	$0.013 \pm 0.001$ $0.009 \pm 0.001$			$0.672 \pm 0.008$	1.00					$-0.9384 \pm 0.0438$	$2 \pm 20$				
												. – – – – .						
	6	WBT-318	dentine	$0.068 \pm 0.001$		13	$0.679 \pm 0.004$	1.00	$883 \pm 110$	$39 \pm 5$	$75 \pm 9$	$76.47 \pm 2.72$	$-0.9016 \pm 0.0479$	$20\pm32$	$89 \pm 12$	$470\pm23$	$579 \pm 41$	$132\pm8$
			enamel	$0.070 \pm 0.001$	$1.276 \pm 0.005$	344	$0.687 \pm 0.003$	1.00					$-0.9386 \pm 0.0461$					

For the Caours samples, the US-ESR model cannot be applied due to high 230Th/234U ratios higher to unity and relatively low equivalent doses, between 50 and 80 Gy. As a consequence, only the AU model was used to calculate ESR/U-series ages, indicating an initial U-uptake systematically followed by a U-leaching for quite all tissues of the five samples. This work demonstrates also the importance of the in situ dosimetry in the age calculation.

homogeneous, leading to a mean age for level 1 of  $124 \pm 10$  ka  $(\pm 2\sigma)$ , similar to the age 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 well confirmed with a mean age of  $125 \pm 9$  ka.

In the Waziers teeth, we observe a very specific geochemical behavior for all the samples, with very low uranium contents and often higher in the enamels than in the dentines. Consequently the internal dose has a restricted impact in the age determination (<10%) compared to the external dose.

The equivalent doses are low but very homogeneous for the N2 teeth, , leading to a mean age for this layer of  $128 \pm 20$  ka ( $\pm 2\sigma$ ), while the DE value obtained for the N6 leads to a Despite all these quite negative points, the obtained ages for N1 teeth are relatively 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 can be calculated from these four samples, confirming the attribution of the deposit to the Eemian interglacial and even, in agreement with the

#### Conclusions

The combined ESR/U-series analyses realized on teeth carried out from two Eemian sites of Northern France, Caours and Wasiers, demonstrate the reliability of the method to date Middle Paleolithic sites and, despite particular geochemical characteristics of part of the analyzed tissues, the validity of the used analytical protocols. This work demonstrates in particular the importance of the in situ dosimetry in the age calculation.

At Caours, the obtained ESR/U-series ages are in excellent agreement with the other available geochronological data derived from U-series and luminescence studies. They permit to calculate new mean age for the lower part of the sequence (124  $\pm$  8 ka for unit 5 (N1, n=7) and 127  $\pm$  15 ka for units 6-11 (including N3, n=6). Associated to available paleo-environmental and archaeological data, they make of this site one of the best known Middle Palaeolithic sequence of France.

For Waziers, the new established ESR/U-series chronology, with a mean age of  $129 \pm 11$  ka, confirms the record of Saalian Late Glacial deposits at the bottom of the sequence and of the subsequent Eemian interglacial stage, evidenced by malacological and palynological studies.

These results show the interest to integrate ESR/U-series dating analyses during the chronostratigraphical study of Middle Paleolithic sites when possible and demonstrate the necessity to involve geochronologists to the fieldworks and excavations, since its earliest stages.

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