

ESR and ESR/U-series chronology of the Middle Pleistocene site of Tourville-la-Rivière (Normandy, France) - A multi-laboratory approach

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1	ESR and ESR/U-series chronology of the Middle Pleistocene site of Tourville-la-Rivière (Normandy, France) - a
2	multi-laboratory approach
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25 26 27 28 29	Abstract – Tourville-la-Rivière (Normandy, France) is one of the rare Middle Pleistocene palaeoanthropological localities of Northern France. Electron Spin Resonance (ESR) and combined ESR/U-series dating methods were independently applied by different teams on sediments and teeth from this site. The present work provides an overview of this multi-laboratory dating work by integrating a description and discussion of the methodologies employed and results obtained.
30 31 32 33	Results confirm that the ESR/U-series analyses of the teeth are greatly dependent on the U-uptake histories of the dental tissues. Although all teeth come from the same archeological level, the samples analysed by each team display two different patterns for the U-series data. This is most likely related to the different sampling areas selected by each team and may be interpreted as the result of local variations in the geochemical

33 areas selected by each team and may be interpreted as the result of local variations in the geochemical 34 conditions of the surrounding environment. Concerning the ESR dating of optically bleached quartz grains, the 35 use of the multiple centre approach seems crucial when dating such fluvial and fluvio-lacustrine sediments. Our 36 results also confirm the great potential of the Ti-H centre to date late Middle Pleistocene deposits.

Despite some (expected) discrepancies related to the independent use of parameters and approaches by the
 different teams involved in this multi-laboratory study, the whole ESR and ESR/U-series data set collected from
 Tourville-la-Rivière locality consistently correlates stratigraphic levels D1 to I and associated human occupation
 to MIS7.

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Keywords – Electron Spin Resonance dating; combined ESR/U-series dating; Tourville-la-Rivière; Middle
 Pleistocene; teeth; optically bleached quart grains.

44 **1.** Introduction

45 Since the late 1980s, Electron Spin Resonance (ESR) of optically bleached quartz grains and combined ESR/U-

46 series of fossil teeth are amongst the most employed dating applications to constrain the chronology of Middle

- 47 Pleistocene archaeo-palaeontological sites. Previous cross-comparison studies showed good agreement with
- 48 results derived from other numerical dating methods such as Luminescence, U-series and ⁴⁰Ar-³⁹Ar (e.g. Duval 49 et al., 2017; Méndez-Quintas et al., 2018; Pereira et al., 2018). Unfortunately, given the very limited number of
- 50 laboratories and researchers specialized in the ESR dating applications mentioned above, inter-laboratory
- 51 comparison studies remain very rare. Additionally, the material analyzed is typically quite small and
- 52 heterogeneous, which does not facilitate the implementation of such large-scale comparative programs.

53 Sometimes, a given site or archaeological level has been dated with ESR or ESR/U-series methods by different 54 teams in separate works, such as for example the sites of La Micoque (Dordogne, France) (Schwarcz and Grün, 55 1988; Falguères et al., 1997) or Atapuerca Gran Dolina TD6 (Spain) (Falguères et al., 1999; Duval et al., 2018) or, 56 more rarely, within the framework of a single study (e.g. Dirks et al., 2017). However, except for critical 57 evaluations of previously published data, these studies have in most cases not led to any proper scientific 58 discussions about the experimental conditions employed and their impact on the final age estimates.

59 Tourville-la-Rivière is one of the rare Middle Pleistocene palaeoanthropological localities of Northern France. It 60 has recently been submitted to a series of independent dating studies by different ESR dating laboratories 61 (CENIEH-RSES, MNHN and BRGM) involving combined ESR/U-series dating of fossil teeth (CENIEH-RSES in Faivre 62 et al., 2014; MNHN in Bahain et al., 2015) and ESR dating of optically bleached quartz grains (MNHN and BRGM, 63 unpublished data). The present paper aims to compile all the chronological data collected for this site in order 64 to enable a proper comparison of the different methodologies employed (from sampling to age determination) 65 and evaluate their impact on the age results. When necessary, new age calculations were performed in accordance to recent methodological developments. These data will contribute to refine the chronology of the 66 67 different archaeological and geological levels for this key palaeoanthropological locality.

68 2. Tourville-La-Rivière site

69 Located on a low fossil fluvial terrace of the Seine System, close to Rouen city in Normandy (Figure 1), Tourville-70 la-Rivière site (Seine-Maritime, France) has been known since the late 1960s (see Lautridou, 1985 and Jamet, 71 2015 and references therein). Several areas within the site have been successively excavated during the 72 following fifty years, delivering an abundant late Middle Pleistocene mammal fauna (Auguste, 2009; Bemilli 73 2010, 2014), a rich archaeological Middle Paleolithic lithic assemblage (Cliquet et al., 2010; Faivre et al., 2014) 74 and three human arm bones attributed to an individual of the Neanderthal lineage (Faivre et al., 2014).

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- 76
- Figure 1 Location of the Tourville-la-Rivière site, Northern France, in the Seine valley terrace system (after 77 Jamet, 2015)

78 Tourville-la-Rivière offers one of the longest Middle Pleistocene continental stratigraphic sequences (>30 m 79 thick) in Western Europe (Lautridou, 1985) (Figure 2). At least two climatic cycles are recorded on a fluvial 80 terrace level corresponding to the T2 terrace of the Seine system (Figure 1). The lower part of the sequence 81 (units A-D) consists of periglacial coarse sands and gravels units with intercalations of fine-grained interglacial 82 fluvial or estuarine sediments. The upper part (units E to J) corresponds to successions of fine-grained 83 sediments, gravels and paleosols, covered by periglacial slope deposits (units K and L) (Lautridou, 1985; Jamet, 84 2015 ; Chauhan et al., 2017). Chronological data available before 2014 (ESR on mollusk shells, Stremme, 1985; 85 Amino acid racemization (AAR) also on mollusk shells, Ochhietti et al., 1987; thermoluminescence (TL) and 86 Infra-red stimulated luminescence (IRSL) on sediments, Balescu et al., 1997; see data in Table 2) correlated the 87 fluvial and estuarine deposits to the Saalian stage and place the deposition of units B and D1 during MIS 9 and 88 7, respectively.

89 The locality has recently been the subject of two successive archaeological excavation campaigns in 2008 90 (Cliquet et al., 2010) and 2010 (Faivre et al., 2014) and a new stratigraphic study was performed within the 91 framework of Guillaume Jamet's PhD thesis (Jamet, 2015). The latter enabled to propose that the position of 92 the end of the MIS7 is recorded higher up in the sequence, i.e. between units E and F instead of between D1 93 and D2 as previously proposed by Lautridou (1985) (Figure 2).

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95 96 97	Figure 2 – Overview of the sedimentary sequence at Tourville-Ia-Rivière, including stratigraphic subdivision, identified biomarkers, numerical dating results available before the recent ESR/U-series and ESR studies, and palaeoenvironmental interpretation (after Jamet, 2015)
98	3. Electron Spin Resonance (ESR) dating: basic principles
99	a. Combined ESR/U-series dating of fossil teeth
100 101 102	The combined use of electron spin resonance (ESR) and U-series dating methods (ESR/U-series) to date Pleistocene mammal remains has been first proposed at the end of 1980s (Grün et al., 1988). The age calculation for a tooth requires the determination of two parameters: an estimate of the total dose of radiation

received during its archaeological history, usually named equivalent dose (D_e), and the dose rate (d_a), i.e. an estimate of the dose annually absorbed by the sample. The D_e value is classically determined using a multiple aliquot additive dose (MAAD) method. The dose rate is assessed from the radioelement content of the sample itself and of the surrounding sediment, in addition to a component from the cosmic rays (sees Duval, 2015 for further detail).

108 The main complication in combined ESR/U-series dating of fossil teeth is related to the uranium incorporation 109 into dental tissues during the fossilization process. This phenomenon depends on the considered tissue, of the 110 geological nature of the site and of its age. It requires the use of mathematical models allowing the description 111 of the U-content evolution with time in a given tissue. The most popular one, uranium-series (US) model, has 112 been proposed by Grün et al. (1988), who introduced a parameter (p) to describe the U-uptake kinetics for 113 each dental tissue. This kinetics is mathematically assessed from the ESR and present-day U-series data 114 measured in each dental tissue. Consequently, only one combined US-ESR age fits the available dataset (see 115 Shao et al., 2015 for the mathematical basis of the US model).

The US model can only be applied if the ESR age calculated assuming an early U-uptake (EU) for all the tissues of a tooth is greater than the corresponding EU-U-series ages. In other words, the occurrence of U-leaching would preclude the use of the US model, which may frequently occur in Pleistocene open air sites. More recently, Shao et al. (2012) proposed the accelerated uptake (AU) model in order to enable combined Useries/ESR age calculations in presence of uranium leaching.

Another alternative to the US model is the closed system uranium-series (CSUS or CSUS-ESR) model that assumes that all uranium migrated into the dental tissue at the time given by the apparent closed system Useries age (Grün 2000). On a given data set, the CSUS model typically provides a maximum possible age estimate. By using both CSUS and US models for a given sample, the resulting age range typically encompass all possible U-uptake histories (e.g. Duval et al., 2018), as long as there are no U-leaching episodes.

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b. ESR dating of optically bleached quartz grains

127 Unlike for fossil teeth, ESR dating of quartz grains is based on the evaluation of light-sensitive signals. The 128 exposure of quartz grains to natural sunlight (an especially to UV-rays; Tissoux et al., 2007) leads to the 129 significant decrease of the intensity associated to the ESR signal some paramagnetic centres. This 130 phenomenon, called optical bleaching, corresponds to a drain of trapped electrons in relation to the energy 131 received during the light exposure (Toyoda et al., 2000). Hence, the event tentatively dated here is not crystal 132 formation, as for the speleothems, or a biological event, as for palaeontological remains, but the last exposure 133 of the quartz grains to the sunlight before their burial into the sediment (Yokoyama et al. 1985).

134 However, the different ESR centres measured in guartz do not all show the same bleaching features. If the ESR 135 intensity of the titanium (Ti) centres (mainly Ti-H and Ti-Li) can be fully reset, the aluminium (AI) signal cannot 136 be zeroed instead. Its ESR intensity decreases until a plateau value corresponding to the presence in the quartz 137 of traps that cannot be emptied by light exposure (Toyoda et al., 2000; Tissoux et al., 2012). This residual value 138 is sample dependent: it is typically determined by exposing an aliquot of the natural sample to a UV solar 139 simulator(e.g. Voinchet et al., 2003). The ESR intensity corresponding to the non-bleachable part of the ESR Al 140 signals is then subtracted to the ESR signal intensity of the studied sample before any D_e determination (so-141 called total bleach method, Forman, 2000). The resulting D_e value corresponds to the total dose of radiation 142 received by the sample during burial (Voinchet et al. 2004).

143 In order to evaluate the bleaching level achieved by the ESR signals during sediment transport, an approach 144 based on the measurements of AI and Ti signals (multiple centre approach, MC) was first proposed by Toyoda 145 et al. (2000). This MC approach aims to take advantage of the different bleaching kinetics typically observed for 146 the Al, Ti -Li and Ti-H centres. The Ti-H signal is known to be fully reset at a much faster rate than the Ti-Li 147 signal, while the Al signal shows a much slower bleaching kinetics in comparison (Toyoda et al., 2000; Duval et 148 al., 2017). Incomplete bleaching would therefore lead to different burial dose estimates for these three ESR 149 signals, with the Ti-H signal typically providing the smallest dose and the Al signal the biggest one. In this case, 150 the first one would most likely be the closest estimate of the true burial dose absorbed by the sample, whereas the second one would provide a maximum possible estimate (see Duval et al., 2017). However, the systematic 151 152 application of the MC approach is sometimes complicated by the weak ESR signal measured for the Ti-H centre 153 (see Rixhon et al., 2017), which makes it complicated to obtain meaningful results.

154 A few recent applications studies may provide a fair idea of the time range applicability achieved by the MC approach. Ti-H signal has proven to produce age estimates consistent with independent age control from about 155 156 300 ka to 40 ka (e.g., Duval et al., 2017; Kreutzer et al., 2018), while Ti-Li and Al centres may provide accurate 157 age results between about 200 ka and 2 Ma (Beerten and Stesman, 2006; Mendez-Quintas et al., 2018; 158 Sahnouni et al. 2018; Voinchet et al., this issue). For a time period younger than about 40 ka, none of the Al and 159 Ti centres seem to yield accurate age results (e.g., Mendez-Quintas et al., 2018), which indicate the difficulty to 160 detect low dose estimates (<100 Gy) with these signals. Finally, one may not exclude that the lower dating limit 161 of the ESR method may well be beyond 2 Ma, as suggested in first instance by the few existing thermal stability 162 studies (see an overview in Toyoda, 2015). However, it is still unclear whether these laboratory estimates are 163 accurate given the uncertainty involved in the evaluation process and the impossibility to reproduce natural 164 condition during annealing experiments.

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166 4. Material and methods

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4.1. Combined ESR/U-series dating of fossil teeth

Eight fossil teeth from the 2010 excavation (Figure 3) were analyzed by the CENIEH-RSES (Centro Nacional de 168 169 Investigación sobre la Evolución Humana, Spain – Research School of Earth Sciences, Australian National 170 University, Australia) team (see details in Faivre et al., 2014): five of them were found in the lower part of Layer 171 D2 (T1, T2, T4, T5 and T8) and three in the upper part (T3, T6 and T7). Four sediment samples were collected 172 from Layer D2, with three in direct contact with T4, T5 and T7. They were used to derive the external beta and 173 gamma dose rate values. Since the dating analyses started at the end of 2011, i.e. more than one year after the 174 end of the excavation (September 2010), the excavation site could no longer be accessed to carry out in situ 175 gamma dose rate measurements.

176

177 Figure 3 – Sampling location of the analyzed teeth and sediments from the Tourville-la-Rivière site

Six horse teeth from the D2 level excavated in 2008 and associated sediments (Figure 3) have been analyzed by the MNHN (Muséum National d'Histoire Naturelle, France) team following the experimental protocol described in Bahain et al. (2012). *In situ* gamma dose rate evaluation was also performed in 2011 on an outcrop connecting the two main excavation areas. Four measurements were performed within D2 layer in order to evaluate lateral variations of radioactivity.

183 The analytical protocols followed by the two teams are summarized in Table 1. They are quite similar for the 184 sample preparation and D_e determination, but differ for the U-series analyses and some of the ESR parameters 185 used in the age calculation. The main differences may be summarized as follows: (i) external dose rate was 186 determined from laboratory analyses of sediment only for CENIEH-RSES tooth samples, while gamma dose rate 187 of the MNHN samples was derived from in situ measurements ; (ii) Potential Rn losses from the dental tissues 188 was evaluated for the MNHN samples, while equilibrium was assumed for the CENIEH-RSES teeth; (iii) the 189 cosmic dose rate was estimated from the present-day depth by CENIEH-RSES team (i.e.21m, leading to a 190 cosmic dose of 26.8 μ Gy/a), while MNHN used a geological model based on the sediment deposition 191 interpretation from Jamet (2015) (leading to a mean cosmic dose of about 100µGy); (iv) the dose rate

conversion factors from Adamiec and Aitken (1998) and Guérin et al. (2011) were used by MNHN and CENIEH RSES teams, respectively; (v) DATA (CENIEH-RSES)(Grün, 2009) and USESR (MNHN)(Shao et al., 2015) combined
 ESR/U-series age calculation programs were employed.

195

196Table 1 – Comparison of the analytical procedures used by the CENIEH-RSES and MNHN teams for the197combined U-series/ESR dating of fossil teeth.

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4.2. ESR dating of quartz grains

Six sediment samples were collected in 2011 at Tourville-la-Rivière, two from the D1 level (Tourville 5 & 6) and another four from the D2 level (Tourville 1 to 4). These last four samples actually correspond to the *in situ* gamma measurement points used for the MNHN teeth (Figure 3). *In situ* gamma measurements were systematically performed at each sampling point using an Inspector1000 Canberra gamma spectrometer. Gamma dose rate were obtained using the threshold approach. By this approach, the count-rate of the spectrometer, proportional to the gamma dose-rate, is directly determined independently of the radioelements sources (U, Th, K) (see details in Mercier & Falguères, 2007).

Four sediment samples were collected by the BRGM (Bureau de Recherche Géologique et Minière, France) team in 2013 (TVL1301 to 04) from one new section cleaned and studied by Guillaume Jamet during his PhD (Figure 4), located approximately 150m ESE from the MNHN sampling section (Figure 3). One sample was taken from the D1 level (TVL1304) and the other three from I level. Here again, *in situ* gamma spectrometry measurements were systematically performed using an Ortec Digidart LF gamma spectrometer and gamma dose rates derived from the Threshold approach.

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Figure 4 – Sampling location of the 2013 sediments from Tourville-la Rivière analyzed by the BRGM team.

The two teams employed the same preparation and measurement procedure. Quartz grains were extracted using physical and chemical preparation techniques described in Voinchet et al. (2004). Aliquots were irradiated using a panoramic ⁶⁰Co source (Dolo et al., 1996) with 1.25 MeV gamma rays and a dose rate of 200Gy/h. Applied irradiation dose values range from 260 to 12,000 Gy.

221 ESR measurements were performed on 100-200 µm quartz grains. The MNHN team measured the Al signal only 222 in all the quartz samples, whereas the BRGM applied the MC approach on two of the four samples analysed 223 (TVL1302 and TVL1304). The residual (non-bleachable) part of the Al signal was determined after exposing the 224 samples to UV light in a Dr Hönhle SOL2 solar simulator for about 1600 hrs. The light intensity received by each 225 artificially bleached samples was comprised between 3.2 et 3.4.10⁵ Lux. ESR measurements were performed at 226 107 K with an EMX Bruker ESR X-band spectrometer using the experimental conditions outlined by Voinchet et 227 al., (2004). Al-signal intensities were measured from the top of the first peak of the hyperfine structure of the 228 quartz ESR spectra at g = 2.018 and the bottom of the 16th peak at g = 2.002 (Toyoda & Falguères, 2003).

The Ti-Li centre intensity was measured from the bottom of the peak at g = 2.002 (royoda & Pagueres, 2005). The Ti-Li centre intensity was measured from the bottom of the peak at g = 1.913 to the baseline (so called option D in Duval and Guilarte, 2015) and the Ti-H centre signal intensity from the bottom of the doublet at g =1.915 to the baseline (so called option C in Duval and Guilarte, 2015). For each centre, the average and the standard deviation of three repeated measurements were calculated and used for D_e determination.

Dose response curves (DRCs) were obtained by fitting an exponential+linear equation (E+L) function through
 the experimental data points (Duval, 2012; Cordier et al., 2012). Fitting was performed using Microcal
 OriginPro 8, with data weighted by the inverse of the square intensities.

The analytical protocols used by the two teams are compared in Table 2. Sample preparation, dose rate evaluation and age calculation are strictly similar for the two procedures. Both teams used measured water content for dose rate evaluation. The main differences concern: (i) the use of the MC approach by BRGM team; (ii) the maximal dose of irradiation (D_{max}) employed by each team (9,870 Gy for MNHN vs 11,700 Gy for BRGM)'; (iii) the water content considered for the age calculation(5-8% for MNHN and 10-12% for BRGM, which is most likely the result of different weather conditions before and during sampling).

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Table 2 – Comparison of the analytical protocols used by the MNHN and BRGM teams for the ESR dating of the
 Tourville sediments.

- 245
- 246 **5.** Results

247 5.1. Combined ESR/U-series age estimates on teeth

248 5.1.1. CENIEH-RSES

249 A summary of the analytical data and results obtained by the CENIEH-RSES team is given in in Table 3. Three of 250 the teeth from lower D2 (T4, T5 and T8) show the highest apparent U-series ages, which precluded any 251 combined US-ESR age calculation for these samples. In contrast, the tooth samples from the upper D2 show a 252 somewhat distinct pattern. The cement tissues of samples T6 and T7 have apparent U-series ages that are 253 significantly older than the other tissues, which again precluded a straightforward US-ESR calculation. In 254 summary, combined US-ESR age calculation was possible for only 3 of the 8 teeth (see also Faivre et al., 2014). 255 All resulting age estimates are within the one-sigma error range. They are between~174 ka and ~208 ka for 256 teeth T1, T2 (both from lower D2) and T3 from the upper part of D2. The limited age scatter (relative standard deviation of about 9%) may be partially explained by some lateral variations in the sediment radioactivity: the 257 258 external dose rate derived from the different sediment samples collected within D2 varies by about 4%.

Interestingly, the CSUS-ESR estimates are somewhat older but nevertheless consistent with the standard US-ESR ages, indicating thus that the U-uptake modelling has a very limited impact on the final age results. They vary between 188 ± 21 and 236 ± 29 ka (Table 3). Based on the weighted mean US-ESR and CSUS-ESR ages, the best age range estimate for the teeth derives from the error range given by both models, i.e. 183 to 226 ka, which represents the middle part of MIS 7 to the beginning of MIS 6.

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Table 3: U-series and ESR data obtained by the CENIEH-RSES team (modified from Faivre et al., 2014)

267 5.1.2. MNHN

Although the combined US-ESR age results obtained by the MNHN team have been published earlier in Bahain
et al. (2015), the data obtained were not fully given and will therefore be detailed in the present paper (Table
4).

271 Contrary to the earlier dating study by Faivre et al (2014), the use of the US model for the age calculation was 272 possible for all the teeth, indicating more homogeneous U-uptake behaviours and no occurrence of uranium 273 leaching. The teeth analyzed by the MNHN team display relatively similar paleodosimetric parameters: the D_e 274 dispersion is low (from 191 ± 6 to 220 ± 2 Gy, around 6%; Table 4); the p parameters describe early uptake (p 275 values between -0.93 and -0.69, Table 4) for all the tissues; the external environmental dose rate measured in 276 situ within the D2 layer shows a very limited lateral variability (5%), demonstrating thus the homogeneity of the 277 sedimentary environment (a mean value of 417 \pm 21 μ Gy/a was used for the age calculation). Hence, the age 278 results obtained for the six teeth show relatively little scatter, i.e. between 203±13 ka and 249±15 ka. A mean 279 age of 224±11 ka could be calculated using IsoPlot 3.0 software (Ludwig, 2003) for the D2 level, unequivocally 280 placing the deposition of this layer during MIS7, and probably during the first part of this stage. A consistent 281 CSUS-ESR weighted mean age of 241±11 ka was obtained, indicating that the U-uptake modelling has a very 282 limited impact on the final age result, similarly to the other dating results obtained by Faivre et al (2014).

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- Table 4 U-series and ESR data obtained by the MNHN team. Analytical uncertainties are given at the one sigma level. * The depth was estimated from the chronostratigraphic interpretation of Jamet (2015).
- 286
- 287 5.2. ESR on optically bleached quartz grains
- 288 *5.2.1.* MNHN

The radioelement contents, ESR ages and associated dose rate contributions determined for the Tourville-la-Rivière D1 and D2 sediments at MNHN are displayed in Table 5.

Table 5- Radioelement contents, equivalent doses, bleaching rate, dose rate contributions and ESR ages
 obtained for the sediments of the Tourville-la-Rivière D1 and D2 levels dated by the MNHN team. The
 bleaching coefficient represents the relative difference between the ESR intensities of the natural and bleached
 aliquots. Analytical uncertainties are given at the one-sigma level.

296 Even if bleaching coefficients vary within narrow range for the whole set of samples from D2 (from 42 to 51%; 297 Table 5), the equivalent dose values differ by a factor of 2.6, while the dose rate are all around 1100-1200 298 μ Gy/a. Consequently, the resulting ESR ages significantly vary, from 349 ± 30 to 906 ± 70 ka. In comparison, 299 the two ESR ages obtained for the D1 level show a limited scatter, with values ranging from 334 \pm 90 to 390 \pm 300 60 ka. This difference between the two levels is probably related with the deposition environment: D1 level is 301 made of coarser sediments than D2, indicating a deposition along the river bank rather than in the floodplain in high water periods. This may explaine why bleaching appears to be more homogeneous among D1 samples 302 303 compared with D2. Following the principles of the MC approach, all these ESR age results obtained from the 304 measurement of the AI centre only should in first instance be interpreted as maximum possible burial age 305 estimates for D1 and D2 levels.

306 5.2.2. BRGM

The results obtained for the samples processed by the BRGM team are displayed in Table 6. The MC approach was employed for two samples (TVL-1302 and TVL-1304) while only the Al-signal was measured for the two other samples.

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Table 11 – Radioelement contents, equivalent doses, bleaching rate, dose rates contributions and ESR ages
 obtained for the sediments of the Tourville-la-Rivière D1 and I levels analyzed by the BRGM team. Analytical
 uncertainties are given at the one-sigma.

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315 In the D1 level, bleaching coefficients of the Al centre determined by the BRGM team are close to those 316 obtained by the MNHN one (42 vs. 46%). Similarly, the total dose rate values are slightly lower but nevertheless 317 consistent at a one-sigma level. In particular, the in situ gamma dose rate of TVL-1304 is the same as the dose 318 measured by MNHN team in this level (samples Tourville 5 and 6; Table 5). Interestingly, both Al and Ti-Li ESR 319 ages are in close agreement around 980-990 ka, and both significantly older than the results derived from 320 MNHN samples of the same level ($334 \pm 90 & 390 \pm 60$ ka; Table 4). In contrast, Ti-H signal provides a 321 significantly younger age of 243 ± 14 ka. Following the principles of the MC approach, these age differences 322 between AI, Ti-Li and Ti-H centres may simply reflect an incomplete bleaching of the former two signals during 323 sediment transport. Consequently, the Ti-H age result is interpreted as being the closest estimate to the true 324 burial age of the sample. This age is actually highly consistent with those derived from the teeth of level D2 325 above (Tables 3 & 4).

326 In contrast, the three samples from level I display very close total dose rate values (around 500-550 μ Gy/a) and 327 highly scattered D_e estimates from the Al centre ranging from 187 ± 73 Gy to 737 ± 178 Gy. As a consequence, 328 The AI ESR ages vary from 535 ± 22 to 1354 ± 283 ka. The Ti-Li signal measured in sample TVL-1302 displays a 329 significantly younger age estimate (by about 44 %) compared with that of the Al signal. Similarly, the Ti-H signal 330 produced an even younger result (-27% compared with Ti-Li). This pattern is comparable to that observed for 331 TVL-1304: it suggests an incomplete reset of both the Al and Ti-Li signals prior to sediment burial. In accordance 332 with the MC approach, the Ti-H signal most likely provides the closest estimate (236 ± 49 ka) to the true burial 333 age for this sample.

Samples TVL-1304 (level D1) and TVL-1302 (level I) are located at the bottom and top of the local sequence,
 respectively, and bracket the human occupation from level D2. They provide very close Ti-H ESR age results
 (243 ± 14 vs. 236 ± 49 ka). As consequence, no apparent stratigraphic pattern is observed. This may be
 interpreted in first instance as an evidence of relatively rapid sedimentation from level D1 to D2. These results

- allow the correlation of the D1 to I levels to MIS7.
- 339 6. Discussion

The two sets of ESR and U-series data obtained on fossil teeth by the two independent teams involved in the Tourville study are overall in good agreement (Figure 5). However, the CENIEH-RSES samples displays more scattered D_e (Figure 5A) and U-series (Figure 5B) data than MNHN, while uranium concentration values in dental tissues overall vary within the same range (Figure 5C).

344

Figure 5 – ESR/U-series data obtained on the Tourville-la Rivière teeth analyzed by the CENIEH-RSES and MNHN
 teams. A) Equivalent dose values; B) U-series data; C) U-content of the dental tissues; D) ESR/U-series (US and
 CSUS) and corresponding age density probability plots.

348 We can observe in particular that a majority of the CENIEH-RSES teeth recovered during the 2010 excavation 349 are close or beyond of the U-series applicability domain, preventing the use of US model for these teeth (Figure 350 5B). The overall smaller D_e values obtained for most of the teeth (Figure 5A) also constitute an additional 351 limiting factor, which explains why US-ESR ages could only be calculated for three teeth. In comparison, the 352 MNHN teeth recovered during the 2008 excavation provide more homogeneous ESR and U-series data and 353 seem to have experienced relatively simple (and relatively early) U-uptake histories, with p-values ranging 354 between -1 and -0.7. In contrast, CENIEH-RSES have apparently experienced more complex U-uptake, including 355 U-leaching. This difference could be correlated to the origin of the teeth, with one excavation area (2008) that 356 underwent more favorable geochemical conditions from an ESR dating perspective in comparison with the 357 other (2010 area). This hypothesis is supported by the fact that two of the three CENIEH-RSES teeth that could 358 be dated are spatially located the closest to the 2008 excavation area (Figure 3).

The MNHN tooth samples yield a weighted mean US-ESR and CSUS-ESR age of 224±11 ka and 241±11 ka, respectively. CENIEH-RSES results are both ~14% lower with 194 $^{+14}_{-11}$ ka and 211 ± 15 ka for the US and CSUS models, respectively. Nevertheless, both data sets overall at a two-sigma confidence level, supporting thus their consistency. Consequently, US-ESR and CS-US-ESR weighted mean ages of 218 ± 16 ka and 236 ± 16 ka may be calculated, respectively (Figure 5D). These results permit to unambiguously correlate the human occupation of the D2 level to MIS 7.

Although both independent data sets are consistent, we acknowledge that there may be a systematic component in the uncertainty due to differences in the analytical procedures, which might partly explain this overall 14% age underestimation of the CENIEH-RSES vs MNHN age results. This age underestimation may be the result of either a D_e underestimation or a dose rate overestimation, or possibly a combination of both.

369 For example, the impact of the absence of in situ gamma dose rate evaluation for the CENIEH-RSES samples can 370 hardly be evaluated. If the impact of the water contents used for age calculations is low (less than 2%) for both 371 dental tissues and sediment, it could be envisaged in first instance that a somewhat overestimated gamma 372 dose rate has been derived from the laboratory analysis of unrepresentative (at a gamma-ray scale) sediment 373 samples. However, the gamma+cosmic dose rate estimated for 2/3 CENIEH-RSES samples are already 374 significantly lower than those obtained for the MNHN samples, mainly in relation to the cosmic dose rate used 375 for the age calculation (26.8 μ Gy/a for the CENIEH-RSES samples vs 100 μ Gy/a for the MNHN ones). 376 Consequently, an additional overestimation of this parameter may be considered as unlikely.

The impact of using different dose rate conversion factors (Adamiec and Aitken (1998) and Guérin et al. (2011) for MNHN and CENIEH-RSES, respectively) is typically estimated to be <1% (e.g., Liritzis et al., 2013), and can therefore here considered to be negligible. Finally, any bias induced by the use of different combined ESR/Useries age calculation programs is also to be negligible (< 1%), as shown earlier in the comparison study by Shao et al. (2014).

382 Additionally, the De values initially considered by Faivre et al (2014) and Bahain et al. (2015) have been 383 recalculated based on the more recent work by Duval and Grün (2016) for the selection of the maximum 384 applied dose (D_{max}) in order to avoid D_e overestimation. Based on their recommendations, and given the 385 magnitude of the D_e values (between 100 and 250 Gy), the D_{max} / D_e ratio should be somewhere of between 5 386 and 10, whereas it is ranging from 20 to 41 for the previously published data (see Table 7). Consequently, new 387 fittings were performed with the same program (Origin), function (SSE), data weighing option $(1/I^2)$ and the 388 appropriate D_{max}/D_e ratio. The resulting D_e values remain all within error, although a slight mean decrease of 389 about 3% of about 6% may be observed for the CENIEH-RSES and MNHN data sets, respectively. One may note

that the errors on the revised D_e values are overall higher than those obtained earlier, which is simply the result of a fitting performed with a more reduced number of experimental data points (6-8 instead of 10 previously). In summary, the use of a smaller D_{max} value has a very limited impact on the corrected D_e value for most of the teeth, and is most likely not the main reason for the overall age difference of ~14% between the two data sets.

In fact, most of this age difference probably comes from the consideration of radon loss from dental tissues by the MNHN team, while a radioactive equilibrium was assumed by the CENIEH-RSES team. The Rn loss measured on both dentine and enamel of the MNHN teeth is quite important for the most of the analyzed tissues (²²²Rn/²³⁰Th ratio ranging from 0.247 to 0.523, except for two tissues showing equilibrium, Table 4). If equilibrium was assumed for these tissues, this would lead to a decrease of the age estimates ranging from -20.7 to -5.8% depending on the samples considered. Moreover, it would no longer be possible to use the US model for two samples (TVL 160 and TVL 923).

401 Lastly, if the MNHN ages are recalculated using the CENIEH-RSES parameters (conversion factors from Guérin 402 et al.(2011) same water contents of dental tissues and sediments, Rn equilibrium, cosmic dose corresponding 403 to a 21 m-depth, gamma dose calculated from the sediment contents), the age estimates decrease for four 404 samples (-12.0 to -13.8 %) and increase for two of them (+1.2 to +8.0 %) leading to a reduced mean age 405 difference of -7.8% between the two sets of samples.

406

407 Table 7 – Comparison of the equivalent dose values obtained with or without use of the recommendations by
 408 Duval and Grün (2016).

409

410 Concerning ESR dating of quartz, all the Al and Ti-Li ESR ages look strongly overestimated (in perhaps a lesser 411 extent for the Ti-Li) compared with the existing chronological framework, the combined ESR/U-series ages and 412 Ti-H ESR ages (Figure 6). In accordance with the principles of the MC approach, this overestimation is most 413 likely due to incomplete bleaching (Duval et al., 2017). The geological nature of the D2 sediment (silty clayey 414 deposits) could indicate a turbid water deposit in a decantation environment such as floodplain or muddy 415 supratidal area. It could explain the age overestimate for Al and Ti-Li centres in this level (see also Voinchet et 416 al., 2015). A similar hypothesis can be made for sandy levels D1 and I. The fluvio-estuarine origin of the 417 sediments seems to constitute an unfavorable environment to completely reset the Al and Ti-Li signals.

In comparison, given its bleaching kinetics, Ti-H centre is by definition most likely to have been fully reset during sediment transport. Ti-H ESR results (243 ± 14 ka and 236 ± 49 ka for units D1 and I respectively) in close agreement with the US-ESR ages obtained on fossil teeth (weighted mean age of 218 ± 16 ka). All together, these data consistently date the deposition of the Tourville D1 to I units to the first part of MIS 7 rather than to the end of this interglacial stage as previously considered (Lautridou, 1985; Balescu et al., 1997). These results demonstrate the importance of using the MC approach in ESR dating of optically bleached quartz grains, which is the only way to evaluate potential incomplete bleaching of Al and Ti centres prior to burial.

- 425
- 426 Figure 6 ESR ages obtained on the Tourville-la Rivière sediments analyzed by the MNHN and BRGM teams
 427 using different quartz ESR centres. The grey band corresponds to the MIS 7 time range.
- 428
- 429 7. Conclusion

430 Tourville-la-Rivière is one of the very few Pleistocene localities where successive and independent ESR and 431 ESR/U-series dating studies have been performed by different teams. The ESR and U-series analyses of fossil 432 teeth show two different populations of samples with different characteristics: the MNHN samples from the 433 2008 excavation are display homogeneous ESR and U-series data and seem to have experienced relatively 434 simple U-uptake histories, while the scattered results obtained on CENIEH-RSES teeth (2010 excavation area) 435 indicate a more complex evolution, including uranium leaching processes. Despite some differences in the 436 analytical protocols independently used by each team, combined ESR/U-series age results consistently position 437 the palaeontological remains and lithic series of D2 layer within MIS7.

- 438 Concerning the ESR dating of optically bleached quartz grains, the two studies performed by the MNHN and 439 BRGM teams show clearly that the sedimentary environments from the clayey unit D2, and the sandy units D1
- and I, were simply not suitable to completely reset the ESR signals of the Al and Ti-Li centres. In contrast, the Ti-
- 441 H centre provides age estimates that are in agreement with the ESR/U-series results. This demonstrates the
- great potential of this centre to date late Middle Pleistocene deposits, which is consistent with previous
- observations by Duval et al. (2017). A multiple centre approach seems therefore indispensable when dating this
- 444 type of fluvio-estuarine sediment, even when the sedimentological characteristics of the sediments seem 445 initially quite suitable for an ESR study (as it was the case for the Tourville D1 and I units). Similar observations
- have been recently made on fluvial deposits from Spain (Duval et al., 2017; Méndez-Quintas et al., 2018), Italy
- 447 (Pereira et al., 2015, 2018; Voinchet et al., this issue) or France (Duval et al., submitted).
- 448 Despite some (expected) discrepancies related to the independent use of parameters and approaches by the
- different teams involved in this multi-laboratory study, the whole set of ESR and ESR/U-series data collected at
- 450 Tourville-la-Rivière locality consistently correlates stratigraphic levels D1 to I and associated human occupation
- 451 to MIS7.

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467 <u>References</u>

- 468 Adamiec, G., Aitken, M., 1998. Dose-rate conversion factor: update. Ancient TL 16, 37-50.
- 469 Auguste P., 2009 Evolution des peuplements mammaliens en Europe du nord-ouest durant le Pléistocène
 470 moyen et supérieur. Le cas de la France septentrionale. Quaternaire, 20, 527-550
- 471 Bahain, J.-J., Falguères, C., Shao, Q., Tombret, O., Duval, M., Dolo, J.-M., 2015. La datation ESR/U-Th de restes
 472 paléontologiques, un outil pour estimer le degré de remaniement des niveaux archéologiques ?
 473 Quaternaire, 26, 213-223
- Bahain, J.-J., Falguères, C., Laurent, M., Shao, Q., Dolo, J.-M. Garcia, T., Douville, E., Frank, N., Monnier, J.-L.,
 Hallegouët, B., Laforge, M., Huet, B., Auguste, P., Liouville, M., Serre, F., Gagnepain, J., 2012. ESR and
 ESR/U-series dating study of several middle palaeolithic sites of Pléneuf-Val-André (Brittany, France): Piégu,
 Les Vallées and Nantois. Quaternary Geochronology, 10, 424-429
- Bahain, J.J., Yokoyama, Y., Falguères, C., Sarcia, M.N., 1992. ESR dating of tooth enamel: a comparison with K-Ar
 dating. Quaternary Science reviews, 11, 245-250.
- Balescu, S., Lamothe, M., Lautridou, J.-P., 1997 Luminescence evidence for two Middle Pleistocene interglacial
 events at Tourville, northwestern France. Boreas, 26, 61-72.
- 482 Beerten, K. and Stesmans, A. (2006). The use of Ti centers for estimating burial doses of single quartz grains: A
 483 case study from an aeolian deposit -2Ma old. Radiation Measurements 41(4): 418-424.
- Bemilli, C. 2010. Les restes fauniques : presentation du corpus et méthodologie, In: Cliquet, D., (dir), 2010 Tourville-la-Rivière, Seine-Maritime. Carrières et ballastières de Normandie : La Fosse-Marmitaine. Section
 BD n° 24 à 30 et 32. INRAP Grand-Ouest, Caen, 105 p (unpublished).

- Bemilli, C., 2014. Les restes fauniques : presentation du corpus et méthodologie, In: Faivre, J.-Ph., (dir), 2014 Tourville-la-Rivière, La Fosse-Marmitaine : une fenêtre sur la vallée de la Seine au cours du Pléistocène
 moyen récent, INRAP Grand-Ouest, Grand-Quevilly, 357 p (unpublished).
- 490 Brennan, B. J., 2003, Beta doses to spherical grains. Radiation Measurements 37, 299-303.
- Brennan, B.J, Lyons, R, Phillips, S. 1991. Attenuation of alpha particle track dose for spherical grains. Nuclear
 Tracks Radiational Measurements 18, 249-253.
- 493 Brennan, B.J., Rink, W.J., McGuirl, E.L., Schwarcz, H.P., Prestwich, W.V., 1997. Beta doses in tooth enamel by 494 "One Group" theory and the Rosy ESR dating software. Radiation Measurements 27, 307–314.
- Chauhan, P. R., Bridgland, D. R., Moncel, M.-H., Antoine, P., Bahain, J.-J., Briant, R., Cunha, P., Despriée, J.,
 Limondin-Lozouet, N., Locht, J.-L., Martins, A., Schreve, D. C., Shaw, A.D., Voinchet, P., Westaway, R., White,
 M. J., White, T. S., 2017. Fluvial deposits as an archive of early human activity: progress during the 20 years
 of the Fluvial Archives Group. Quaternary Science Reviews, 166, 114-149
- Cliquet, D., (dir), 2010 Tourville-la-Rivière, Seine-Maritime. Carrières et ballastières de Normandie : La Fosse Marmitaine. Section BD n° 24 à 30 et 32. INRAP Grand-Ouest, Caen, 105 p (unpublished).
- Cordier, S., Harmand, D., Lauer, T., Voinchet, P., Bahain, J.-J., Frechen M., 2012. Geochronological
 reconstruction of the Pleistocene evolution of the Sarre valley (France and Germany) using OSL and ESR
 dating techniques. Geomorphology, 165–166, 91–106
- Dolo, J.-M., Lecerf, N., Mihajlovic, V., Falguères, C., Bahain, J.-J., 1996. Contribution of ESR dosimetry for
 irradiation of geological and archaeological samples with a 60-Co panoramic source, Applied Radiation and
 Isotopes 47, 1419-1421.
- Dirks, P.H.G.M., Roberts, E.M., Hilbert-Wolf, H., Kramers, J.D., Hawks, J., Dosseto, A., Duval, M., Elliott, M.,
 Evans, M., Grün, R., Hellstrom, J., Herries, A.I.R., Joannes-Boyau, R., Makhubela, T.V., Placzek, C.J., Robbins,
 J., Spandler, C., Wiersma, J., Woodhead, J. and Berger, L.R. (2017). The age of Homo naledi and associated
 sediments in the Rising Star Cave, South Africa. eLife 6: e24231.
- 511 Duval, M. (2012). Dose response curve of the ESR signal of the Aluminum centre in quartz grains extracted from
 512 sediment. Ancient TL 30(2): 1-9.
- 513 Duval, M. (2015). Electron Spin Resonance (ESR) Dating of Fossil Tooth Enamel. Encyclopedia of Scientific
 514 Dating Methods. W. J. Rink and J. W. Thompson, Springer Netherlands: pp 239-246.
- 515 Duval, M., Arnold L.J., Guilarte V., Demuro M., Santonja M, Pérez-González, A., 2017. Electron spin resonance
 516 dating of optically bleached quartz grains from the Middle Palaeolithic site of Cuesta de la Bajada (Spain)
 517 using the multiple centres approach. Quaternary Geochronology 37, 82-96.
- 518 Duval, M., Grün, R., Falguères, C., Bahain, J.J., Dolo, J.M., 2009. ESR dating of Lower Pleistocene fossil teeth:
 519 Limits of the single saturating exponential (SSE) function for the equivalent dose determination. Radiation
 520 Measurements 44, 477-482.
- Duval, M., Grün, R., Parés, J.M., Martín-Francés, L., Campaña, I., Rosell, J., Shao, Q., Arsuaga, J.L., Carbonell, E.
 and Bermúdez de Castro, J.M. (2018). The first direct ESR analysis of a hominin tooth from Atapuerca Gran
 Dolina TD-6 (Spain) supports the antiquity of Homo antecessor. Quaternary Geochronology 47, 120-137.
- Duval, M., Guilarte, V., 2015. ESR dosimetry of optically bleached quartz grains extracted from Plio-quaternary
 sediment: evaluating some key aspects of the ESR signal associated to the Ti-centre. Radiation
 Measurement 78, 28–41
- Duval M., Voinchet P., Arnold L.J., Parés J.M., Minnella W., Guilarte V., Demuro M., Falguères C., Bahain J.-J. &
 Despriée J. (submitted). A multi-technique dating study sheds new light on the chronology of two Lower
 Palaeolithic sites of the Middle Loire Basin, France: Lunery-la Terre-des-Sablons and Brinay-la Noira.
 Submitted to Quaternary International.
- Faivre, J.-P., Maureille, B., Bayle, P., Crevecoeur, I., Duval, M., Grün, R., Bemilli, C., Bonilauri, S., Coutard, S.,
 Bessou, M., Limondin-Lozouet, N., Cottard, A., Deshayes, T., Douillard, A., Henaff, X., Pautret-Homerville, C.,
 Kinsley, L., Trinkaus, E., 2014 Middle Pleistocene human remains from Tourville-la-Rivière (Normandy,
 France) and their archaeological context. PlosOne, 9 (10), e104111.
- Falguères, C., Bahain, J.-J., Saleki, H., 1997. U-series and ESR dating of teeth from Acheulian and Mousterian
 levels at La Micoque (Dordogne, France). Journal of Archaeological Science, 24, 537-545
- Falguères, C., Bahain, J.-J., Yokoyama, Y., Arsuaga, J.L., Bermudez De Castro, J.M., Carbonell, E., Bischoff, J.L.,
 Dolo, J.M., 1999. Earliest humans in Europe: the age of TD6 Gran Dolina, Atapuerca, Spain. Journal of

- 539 Human Evolution, 37, 343-352.
- Forman, S. L., J. Pierson and K. Lepper 2000. Luminescence Geochronology. Quaternary Geochronology:
 methods and applications. J. Sowers, J. Noller and L. WR. Washington, DC, American Geophysical Union:
 157-176.
- 543 Grün, R, Katzenberger-Apel, O., 1994. An alpha irradiator for ESR dating. Ancient TL 12, 35-38.
- 544 Grün, R. 2000a. An alternative for model for open system U-series/ESR age calculations: (closed system U-545 series)-ESR, CSUS-ESR. Ancient TL 18, 1-4.
- 546 Grün, R. (2000b). Methods of dose determination using ESR spectra of tooth enamel. Radiation Measurements
 547 32(5-6): 767-772.
- 548 Grün, R., 2009. The DATA program for the calculation of ESR age estimates on tooth enamel. Quaternary 549 Geochronology 4, 231-232.
- Grün, R., Schwarcz, H.P., Chadam, J.M., 1988. ESR dating of tooth enamel: coupled correction for U-uptake and
 U-series disequilibrium. Nuclear Tracks and Radiation Measurements, 14, 237-241.
- Grün, R., S. Eggins, L. Kinsley, H. Moseley and M. Sambridge (2014). Laser ablation U-series analysis of fossil
 bones and teeth. Palaeogeography, Palaeoclimatology, Palaeoecology 416: 150-167.
- 554 Guérin, G., Mercier, N., Adamiec, G., 2011. Dose-rate conversion factors: update. Ancient TL 29, 5-8.
- Jamet, G., 2015. Réponses sédimentaires d'un bassin versant côtier aux variations glacio-eustatiques et au
 soulèvement plio-quaternaire : l'exemple du bassin versant côtier de la baie de Seine (Seine, Touques et
 Dives). PhD thesis, University of Caen, 418p (unpublished)
- Kreutzer, S., Duval, M., Bartz, M., Bertran, P., Bosq, M., Eynaud, F., Verdin, F. and Mercier, N. (2018).
 Deciphering long-term coastal dynamics using IR-RF and ESR dating: a case study from Médoc, south-west
 France. Quaternary Geochronology 48: 108-120.
- Lautridou, J.-P., 1985. Le cycle périglaciaire pléistocène en Europe du Nord-Ouest et plus particulièrement en
 Normandie. State doctorate thesis, Centre de Géomorphologie du CNRS éd., Caen, 907 p.
- Liritzis, I., Stamoulis, K., Papachristodoulou, C. and Ioannides, K. (2013). A re-evaluation of radiation dose-rate
 conversion factors. Mediterranean Archaeology and Archaeometry 13(3): 1-15.
- Ludwig, K. R., 2003. Isoplot 3.0, a geochronological toolkit for Microsoft Excel. Berkeley Geochronology Centre
 Special Publication, 4, 71p
- Marsh, R.E., 1999. Beta-gradient isochrons using electron paramagnetic resonance: Towards a new dating
 method in archaeology. MSc thesis, McMaster University, Hamilton.
- Méndez-Quintas, E., Santonja, M., Pérez-González, A., Duval, M., Demuro, M., Arnold, L. J., 2018. First evidence
 of an extensive Acheulean large cutting tool accumulation in Europe from Porto Maior (Galicia, Spain).
 Scientific Reports 8:3082.
- 572 Mercier, N, Falguères, C, 2007. Field gamma dose-rate measurement with a Nal(Tl) detector: re-evaluation of 573 the "threshold" technique. Ancient TL Vol. 25 No.1
- Occhietti, S., Pichet, P., Rheault, L., 1987. Résultats préliminaires d'aminochronologie moyenne et basse vallée
 de la Seine in Lautridou J.-P., La Normandie, Guide d'excursion de l'Association Française pour l'Étude du
 Quaternaire, additif, 19 p.
- Pereira, A., Nomade, S., Moncel, M.-H., Voinchet, P., Bahain, J.-J., Biddittu, I., Falguères, C., Giaccio, B., Manzi,
 G., Parenti, F., Scardia, G., Scao, V., Sottili, G., Vietti, A., 2018. Geochronological evidences of a MIS 11 to
 MIS 10 age for several key Acheulian sites from the Frosinone province (Latium, Italy): Archaeological
 implications. Quaternary Science Reviews, 187, 112-129
- Pereira, A., Nomade, S., Voinchet, P., Bahain, J.-J., Falguères, C., Garon, H., Lefèvre, D., Raynal, J.-P., Scao, V.,
 Piperno, M., 2015. The earliest securely dated hominid fossil in Italy and evidences of Acheulian human
 occupations during glacial MIS 16 at Notarchirico (Venosa, Basilicata, Italy). Journal of Quaternary Science,
 30 (7), 639-650
- Prescott, J.R., Hutton, J. T., 1994. Cosmic ray contributions to dose rates for Luminescence and ESR Dating:
 Large depths and long-term time. Radiation Measurements. 23, 497-500
- Prescott, J.R., Hutton, J.T., 1988. Cosmic ray and gamma ray dosimetry for TL and ESR. Nuclear Tracks and
 Radiation Measurements 14: 223-227.
- 589 Sahnouni, M., Parés, J.M., Duval, M., Cáceres, I., Harichane, Z., van der Made, J., Pérez-González, A.,

- Abdessadok, S., Kandi, N., Derradji, A., Medig, M., Boulaghraif, K. and Semaw, S. (2018). 1.9-million- and
 2.4-million-year-old artifacts and stone tool–cutmarked bones from Ain Boucherit, Algeria. Science
 362(6420): 1297-1301.
- 593 Schwarcz, H. P., Grün, R., 1988. ESR dating of level L2/3 at La Micoque, Dordogne (France): Excavation of 594 Debénath and Rigaud. Geoarchaeology 3–4, 293–296.
- Shao, Q., Bahain, J.-J., Falguères, C., Dolo, J.-M., Garcia, T., 2012. A new U-uptake model for combined ESR/U series dating of tooth enamel. Quaternary Geochronology 10: 406–411.
- Shao, Q., Bahain, J.J., Dolo, J.M., Falguères, C., 2014. Monte Carlo approach to calculate US-ESR ages and their
 uncertainties. Quaternary Geochronology 22, 99-106.
- Shao, Q., Chadam, J., Grün, R., Falguères, C., Dolo., J.-M., Bahain, J.-J., 2015. The mathematical basis for the US ESR dating method. Quaternary Geochronology 30: 1–8.
- Stremme, H.E., 1985. Altersbestimmung und Palaoboden in der Quatarstratigraphie. Bulletin de l'Association
 Française pour l 'Etude du *Quaternaire*, 2-3, pp.159-166.
- Tissoux, H., Falguères, C., Voinchet, P., Toyoda, S., Bahain, J.J. and Despriée, J. (2007). Potential use of Ti-center
 in ESR dating of fluvial sediment. Quaternary Geochronology 2(1–4): 367-372.
- Tissoux, H., Toyoda, S., Falguères, C., Voinchet, P., Takada, M., Bahain, J.-J., Despriée, J., 2008. ESR dating of
 sedimentary quartz from two Pleistocene deposits using Al and Ti-centres. <u>Geochronometria 30, 23-31.</u>
- Tissoux, H., P. Voinchet, F. Lacquement, F. Prognon, D. Moreno, C. Falguères, J.-J. Bahain and S. Toyoda (2012).
 Investigation on non-optically bleachable components of ESR aluminium signal in quartz. Radiation
 Measurements 47(9): 894-899.
- Toyoda, S. (2015). Paramagnetic lattice defects in quartz for applications to ESR dating. Quaternary
 Geochronology 30, Part B: 498-505.
- Toyoda, S., Falguères C., 2003. The method to represent the ESR signal intensity of the aluminium hole centre
 in quartz for the purpose of dating. Advances in ESR applications, 20, 7-10.
- Toyoda, S., Voinchet, P., Falguères, C., Dolo, J.-M., Laurent, M., 2000. Bleaching of ESR signals by the sunlight: a
 laboratory experiment for establishing the ESR dating of sediments. Applied Radiation and Isotopes 52,
 1357-1362,
- Voinchet, P., C. Falguères, M. Laurent, S. Toyoda, J. J. Bahain and J. M. Dolo (2003). Artificial optical bleaching
 of the Aluminium center in quartz implications to ESR dating of sediments. Quaternary Science Reviews
 22(10–13): 1335-1338.
- Voinchet, P., Bahain, J.-J., Falguères, C., Laurent, M., Dolo, J.-M., Despriée, J., Gageonnet R., 2004. ESR dating of
 quartz extracted from Quaternary sediments: Application to fluvial terraces system of Northern France.
 Quaternaire, 15, 135-141.
- Voinchet, P., Toyoda, S., Falguères, C., Hernandez, M., Tissoux, H., Bahain, J.-J., 2015. ESR residual dose in
 quartz modern samples, an investigation into environmental dependence. Quaternary Geochronology, 30,
 506-512
- Voinchet P., Falguères C., Pereira A., Nomade S., Biddittu I., Pipperno M., Bahain J.-J. (this issue). ESR dating
 applied to optically bleached quartz-a comparison with 40Ar/39Ar chronologies on Italian Middle
 Pleistocene sequences.
- Yokoyama, Y., Falguères, C., Quaegebeur, J.P., 1985. ESR dating of quartz from Quaternary sediments: first
 attempt. Nuclear tracks 10, 921–928.
- 631

632 Figure caption

- Figure 1 Location of the Tourville-la-Rivière site, Northern France, in the Seine valley terrace system (after Jamet, 2015)
- 635 Figure 2 Overview of the sedimentary sequence at Tourville-la-Rivière, including stratigraphic subdivision,
- 636 biomarkers identified, numerical dating results available before the recent ESR/U-series and ESR studies, and 637 palaeoenvironmental interpretation (after Jamet, 2015)
- 638 Figure 3 Sampling location or the analyzed teeth and sediments from the Tourville-la-Rivière site
- 639 Figure 4 Sampling location of the 2013 sediments from Tourville-la Rivière analyzed by the BRGM team.

- 640 Figure 5 ESR/U-series data obtained on the Tourville-la Rivière teeth analyzed by the CENIEH-RSES and MNHN
- teams. A) Equivalent dose values; B) U-series data; C) U-content of the dental tissues; D) ESR/U-series (US and
 CS-US) and corresponding age density probability plots.
- Figure 6 ESR ages obtained on the Tourville-la Rivière sediments analyzed by the MNHN and BRGM teams using different ESR centres. The grey band corresponds to the MIS 7 time range.
- 645

646 Table caption

- Table 1 Comparison of the analytical procedures used by the CENIEH-RSES and MNHN teams for the combined U-series/ESR dating of fossil teeth.
- Table 2 Comparison of the analytical protocols used by the MNHN and BRGM teams for the ESR dating of the
 Tourville sediments.
- Table 3: U-series and ESR data obtained by the CENIEH-RSES team (modified from Faivre et al., 2014).
- Table 4 U-series and ESR data obtained by the MNHN team. Analytical uncertainties are given with $\pm 1\sigma$. * The depth was estimated from the chronostratigraphic interpretation of Jamet (2015).
- Table 5– Radioelement contents, equivalent doses, bleaching rate, dose rate contributions and ESR ages
 obtained for the sediments of the Tourville-la-Rivière D1 and D2 levels dated by the MNHN team. The
 bleaching coefficient represents the relative difference between the ESR intensities of the natural and bleached
 aliquots. Analytical uncertainties are given with ±1σ.
- 658Table 6 Radioelement contents, equivalent doses, bleaching rate, dose rates contributions and ESR ages659obtained for the sediments of the Tourville-la-Rivière D1 and I levels analyzed by the BRGM team. Analytical660uncertainties are given with $\pm 1\sigma$.
- Table 7 Comparison of the equivalent dose values obtained with or without use of Duval and Grün (2016)'s
 recommendations.
- 663

					Apparent	U- In	itial enamel	Removed		Sedi	ment radioele	ment	
Sample	Tissue	U (nnm)	²³⁴ U/ ²³⁸ U	²³⁰ Th/ ²³⁸ U	series ag	ge	Thickness	thickness	Depth (m)		contents	~	D _E
		(ppiii)			(ka)		(µm)	(µm)	(11)	U (maa)	nı (mag)	к (%)	(0y)
	e n e n e l	0.39	1.2266	0.6211	75.4			(1), 100 + 10		<u></u> ,	ur 7	V: 7	
Т1	enamei	± 0.00	± 0.0483	± 0.0331	75.1		1160	(1): 100 ± 10	21	1.11	4.09	0.67	121
11	dentine	19.68	1.2366	0.7922	106.8		± 116	(2): 110 ± 11	± 3	± 0.02	± 0.02	± 0.02	± 2
		± 0.12	± 0.0127	± 0.0471				(_):					
	enamel	0.53 + 0.02	1.2110	1.0357	189.0		1010	(1): 110 ± 11	21	1 1 1	4.00	0.67	175
T2		28.49	1 3172	1 0/15			+ 1010		+ 3	+ 0.02	+ 0.02	+ 0.07	+ 2
	dentine	± 0.97	± 0.0032	± 0.0103	154.0		2 101	(2): 100 ± 10	- 3	10.01	10.02	10.02	÷ •
	onamol	0.42	1.2097	0.7048	02.2			(1): 00 + 0					
тз	enamer	± 0.11	± 0.0383	± 0.1833	92.5		1300	(1). 90 ± 9	20	0.88	3.73	0.72	142
15	dentine	28.19	1.2468	0.8435	116.8		± 130	(2): 120 ± 12	± 3	± 0.02	± 0.02	± 0.02	± 4
		± 1.64	± 0.0070	± 0.0155				()					
	enamel	0.41 + 0.14	1.2398	1.3390	656.2		900	(1): 110 ± 11	21	1 47	2 5 2	0.67	165
T4		24.10	1.3209	1.0818			± 90		+ 3	+ 0.02	± 0.02	± 0.07	± 2
	dentine	± 1.97	± 0.0102	± 0.0175	165.5		200	(2): 90 ± 9		_ 0.02	_ 0.02	- 0.02	
	anomal	0.71	1.3082	1.2751	265.7			(1), 50 + 5)				
Τ5	enamer	± 0.27	± 0.0475	± 0.0742	205.7		970	$(1): 50 \pm 5$	21	1.47	3.52	0.67	155
15	dentine	23.04	1.3608	1.2050	196.1		± 97	(2): 80 ± 8	± 3	± 0.02	± 0.02	± 0.02	± 4
		± 0.93	± 0.0084	± 0.0585									
	enamel	0.41 + 0.12	1.1931	0.8108	118.8			(1): 30 ± 3					
		27.85	1.2029	0.8059			890		21	0.88	3.73	0.72	153
Т6	dentine	± 2.43	± 0.0069	± 0.0067	115.7		± 89		± 3	± 0.02	± 0.02	± 0.02	± 4
	comontum	29.98	1.2836	0.8287	107 E			(2): 60 ± 6					
	cementum	± 6.69	± 0.0059	± 0.0326	107.5			/					
	enamel	0.49	1.2791	0.7844	99.1			(1): 20 ± 2					
		± 0.18	± 0.0578	± 0.1109			1050		21	0 00	2 72	0.72	164
T7	dentine	29.90	+ 0.0080	+ 0.0055	106.2		+ 105		+ 3	0.88	3.73	+ 0.02	104 + 4
		37.26	1.3047	0.7773			105	(2): 60 ± 6	÷J	± 0.02	10.02	± 0.02	± 7
	cementum	± 1.27	± 0.0105	± 0.0124	94.5								
	enamel	0.82	1.3203	1.1941	209.8			(1): 40 + 4					
Т8	chunch	± 0.37	± 0.0437	± 0.1182	205.0		840	(1). 10 = 1	20	0.87	3.77	0.72	254
	dentine	17.76	1.3777	1.3479	259.6		± 84	(2): 60 ± 6	± 3	± 0.02	± 0.02	± 0.02	± 4
		± 0.46	± 0.0089	± 0.0132		· · · · · ·							
	Dental		n I Luntako	D		D	D	D	r	h	FSR-11-corios	<u>(</u>	IS_ESP
Sample	tissue	pa	rameter (a.u.)	(μGy/a		μGy/a)	(μGy/a)	(μGy/a)	- (μG	iy/a)	age (ka)	age	e (ka)
					7								
	enamel		-0.09 ± 0.27										
T1	dentine		-0.51 ± 0.20	56 ± 16		133 ± 33	61 ±11	405 ± 55	656	± 67	184 + 26 - 19	201	L ± 25
	enamel		0.02 + 0.06										
Т2	enamer	-	-0.95 ± 0.00	185 ± 3	3	344 ± 63	68 ± 12	405 ± 52	1003	3 ± 88	174 +17 -14	188	3 ± 21
	dentine	-	-0.93 ± 0.06)									
T 2	enamel		-0.12 ± 0.50	74 + 45		172 + 20	FF 1 40	201 - 40	600	+ 72	200 1 20 22	22/	1.20
13	dentine		-0.49 ± 0.14	/1 ± 40		1/3 ± 30	55 ± 10	381 ± 48	680	± /2	208 ± 28 -22	236) ± 29
									Weighte	d mean	194 + 14 -11	211	! ± 15
			V										
			1										

Sample	Dental tissue	U (pp	m)	²³⁴ U/ ²³⁸ U	²³⁰ Th/ ²³⁸ U	Apparent U-series age (ka)	²²² Rr	ነ/ ²³⁰ Th	tl	Initial enamel nickness (µm)	Removed thickness (μm)	Mean depth (m) *	De (Gy)
TVL 157	enamel dentine	0.594 ± (25.342 ±	0.024 0.580	1.441 ± 0.069 1.306 ± 0.026	1.222 ± 0.101 1.028 ± 0.052	174 153	0. 0.	334 366	ç	959 ± 17	(1) 21 ± 3 (2) 74 ± 9	14 ± 3	220.08 ± 2.33
TVL 160	enamel	0.402 ± (0.016	1.313 ± 0.060	1.155 ± 0.102 1 152 ± 0.049	195	1.	000	10)50 ± 131	$(1) 28 \pm 3$ $(2) 160 \pm 20$	14 ± 3	207.72 ± 2.72
TVL 219	enamel	0.671 ±	0.022	1.353 ± 0.020 1.259 ± 0.043	0.993 ± 0.043	155	0.	405	10)27 ± 128	$(2) 100 \pm 20$ (1) 14 ± 2 (2) 167 + 21	14 ± 3	204.54 ± 2.49
TRV 923	enamel	0.490 ± 0	0.030	1.274 ± 0.022 1.301 ± 0.054	1.106 ± 0.094	140	0.	259 202	9	58 ± 120	$\begin{array}{c} (2) \ 167 \pm 21 \\ \hline (1) \ 68 \pm 9 \\ \hline (2) \ 76 \pm 0 \end{array}$	14 ± 3	204.56 ± 1.09
TRV 928	enamel	0.296 ±	0.741	1.261 ± 0.028 1.409 ± 0.076	1.005 ± 0.058 1.305 ± 0.116	217	0.	293 258	12	268 ± 159	$(2) 76 \pm 9$ (1) 200 ± 25 (2) 148 ± 18	14 ± 3	191.33 ± 5.82
TVL 929(a)	enamel	0.374 ± 0	0.348	1.311 ± 0.021 1.236 ± 0.047 1.263 ± 0.025	0.973 ± 0.085	155	0.	247 000	12	200 ± 150	$(2) 140 \pm 10$ $(1) 112 \pm 14$ $(2) 60 \pm 8$	14 ± 3	191.95 ± 2.73
Sample	Sediment r	radioelemen	t contents	Dental	p U-uptake		D _α	D	ß	D _{(γ+cosm}	D_a	US-ESR a	CSUS- ge ESR age
TVL 157	1.072	4.005	0.894	enamel	-0.83 ± 0.04	.,	139	(μGy 30	//a)	517	961) (ка) 229	(ka) 252
TVL 160	1.252	4.341	0.902	dentine enamel	-0.72 ± 0.05 -0.91 ± 0.03		100	29	4	± 29 517	911	± 13	± 10
TVI 219	± 0.074	± 0.099	± 0.011 0.853	dentine enamel	-0.88 ± 0.03 -0.82 ± 0.05		± 19 142	± 4	.9	± 29 517	± 56	± 13 203	± 12 223
102 213	± 0.076	± 0.102	± 0.012	dentine	-0.73 ± 0.05		± 28	± 5	5	± 29	± 61	± 13	± 14
TRV 923	1.065 ± 0.063	3.716 ± 0.084	0.837 ± 0.010	enamel dentine	-0.88± 0.04 -0.78 ± 0.04		103 ± 21	31 ± 4	4 17	517 ± 29	934 ± 59	219 ± 13	232 ± 14
TRV 928	1.120 ± 0.078	3.735 ± 0.103	0.799 ± 0.012	enamel dentine	-0.93 ± 0.03 -0.69 ± 0.05		75 ± 26	17 ± 3	6 9	517 ± 29	768 ± 55	249 ± 15	268 ± 15
TVL 929(a)	1.151 ± 0.086	3.678 ± 0.114	0.807 ± 0.014	enamel dentine	-0.74 ± 0.04		65 ± 14	27 ± 4	1 3	517 ± 29	853 ± 54	225 ± 13	240 ± 13
											Weigthed med	an 224±1.	1 241 ± 11

Sample	Level	U (ppm)	Th (ppm)	K (%)	Al Bleaching coefficient (%)	D _e (Gy)	D _α (μGy/a)	D _β (μGy/a)	<u>in situ</u> D _γ (μGy/a)	D _{cosmic} (μGy/a)	D _a (µGy/a)	Age (ka)
Tourville 1	D2	1.014 ±	3.270±	0.748±	44	409	26	622	393	102	1144	358
Tourville 2	D2	0.074 0.895 ±	3.133 ±	0.012 0.728 ±	42	± 56 398	±1 24	± 17 595	± 20 418	± 5 102	± 22 1139	± 50 349
	DZ	0.083	0.114	0.014	72	± 30 708	±1 27	± 19 629	± 20 412	± 5 102	± 50	± 30 605
Tourville 3	D2	0.086	0.118	0.014	49	± 87	±1	± 20	± 20	±5	± 26	± 80
Tourville 4	D2	0.990 ± 0.072	3.567 ± 0.099	0.741± 0.012	51	1085 ± 76	27 ±1	621 ±17	447 ± 20	102 ± 5	1198 ± 21	906 ± 70
Tourville 5	D1	0.767±	1.677±	0.426±	46	246 + 22	16 + 1	370 + 10	260 + 12	89 + 4	736	334 + 00
Tourville 6	D1	0.040 0.711±	1.978±	0.000 0.447 ±	46	<u>1 32</u> 295	18	389	260	<u> </u>	756	<u> </u>
	DI	0.067	0.080	0.010	40	± 40	±1	± 15	± 13	± 4	± 19	±60

Sample	Level	U (ppm)	Th (ppm)	К (%)	ESR Centre	Bleaching rate (%)	De (Gy)	D _α (μGy/a)	D _β (μGy/a)	in situ D _γ (μGy/a)	D _{cosmic} (µGy/a)	Da (µGy/a)	Age (ka)
TVL1301	I	0.320 + 0.038	0.822 + 0.043	0.361 + 0.005	Al	37	335 ± 62	9 + 1	307 + 9	176 + 55	47 + 2	538 + 56	565 + 32
		_ 01000	_ 010 10	_ 0.000	Al	43	332 ± 113						577
TVL1302		0.356	0.830	0.347	Tili	100	197 + 72	9	302	163	47	521	± 120 325
	1	± 0.042	± 0.048	± 0.006	11-61	100	107 ± 73	±1	±10	± 55	± 2	± 56	± 68 236
		0.000	0.740		Ti-H	100	136 ± 39		270	170		504	± 49
TVL1303	Ι	0.262 ± 0.039	0.713 ±0.044	0.333 ± 0.005	Al	41	737 ± 178	/ ±1	278 ±9	172 ± 60	47 ± 2	504 ±61	1354 ± 283
					Al	42	687 ± 255						981 ± 204
TVL1304	D1	0.573	1.377	0.400	Ti-Li	100	696 ± 183	14	362	260	38	674	994
		±0.043	±0.051	± 0.006	T : 11	100	170 12	±1	±10	± 54	±2	± 55	± 206 243
					II-H	100	170±13				<u> </u>		± 14
									() /			
									1				
					. ~								
			C										
			Y										
			r										

Sample	CI	ENIEH-RSES		Т	his study		Ratio
	(Faiv	re et al., 201	4)				
	D _e -1 (Gy)	D _{max} (Gy)	Dmax/D _e -1	D _e -2 (Gy)	D _{max} (Gy)	D_{max}/D_e -2	D _e -2/ D _e -1
T1	121 ± 2	5,000	41	121 ± 3	1,000	8	1.00
T2	175 ± 2	5,000	29	178 ± 4	1,000	6	1.02
T3	142 ± 4	5,000	35	134 ± 4	1,000	7	0.94
14	165 ± 2	5,000	30	160 ± 2	1,000	6	0.97
15	155 ± 4	5,000	32	150 ± 7	1,000	7	0.97
	153 ± 4 164 + 4	5,000	33	149 ± 7 151 + 5	1,000	7	0.97
17	104 ± 4 254 ± 4	5,000	20	151 ± 5 251 ± 9	1,000	7	0.92
o h a + a coM	234 ± 4 166 + 28 (17%)	5,000	20	231 ± 0 152 + 77 (1/1%)	1,000	/	0.99
Sample	100 1 20 (1770)	MNHN		<u> </u>	his study		Ratio
Sample	(Baha	ain et al. ,201	.5)		ins study		Natio
	D _e -1 (Gy)	D _{max} (Gy)	Dmax/D _e -1	D _e -2 (Gy)	D _{max} (Gy)	D _{max} /D _e -2	D _e -2/D _e -1
TVL157	221 ± 3	5,000	23	217 ± 7	1,249	6	0.98
TVL160	208 ± 3	5,000	24	206 ± 6	1,249	6	0.99
TVL219c	205 ± 2	5,000	24	193 ± 5	1,249	6	0.94
TVL923	205 ± 1	5,000	24	201 ± 3	1,249	6	0.98
TVL928	191 ± 6	5,000	23	169 ± 6	1,249	7	0.88
TVL929	191 ± 6	5,000	26	183 ± 4	1,249	7	0.96
Mean ± s.d.	207 ± 6 (3%)			195 ± 14 (7%)			0.96
					5		
			, O				

Analytical steps	CENIEH-RSES	MNHN
Preparation	Mechanic with	dentist drill
Selected grain size	100-200) μm
Impact of preparation on alpha and beta dose rate	Data from Marsh (1999)	Data from Brennan et al. (1997)
Number of dose irradiation steps	10	
Maximum irradiation dose (D _{max})	5,000	Gy
Measurement of the ESR intensity	Peak-to-peak amplitude	(T1B2; Grun, 2000b)
Fitting function - data weighting Fitting program	Single Saturating Exponential (SSE) – Inver Origi	rse of the squared ESR intensities (1/l ²) n
U-series analyses of dental tissues	High resolution Laser Ablation ICP-MS analyses (as in Grün et al., 2014)	Solution alpha spectrometry bulk analyses
	The U-series data from all the laser ablation spots of	
	a given tissue were combined to provide the data input for the ESR age calculations	
Alpha efficiency	0.13 ± 0.02 (Grün and Kat	zenberger-Apel, 1994)
Rn loss in dental tissues	Equilibrium was assumed	Determined by cross-checking data from High
		resolution gamma spectrometry (HRGS) and alpha spectrometry analyses
Water content of dental tissues	Enamel = 0 % wgt	Enamel = 0 % wgt
	Dentine = 5 ± 3 % wgt	Dentine = $7 \pm 5\%$ wgt
Radioelement contents in sediment	Determined by ICP-MS/OES	Determined by HRGS
	The values were used to derive the beta and gamma	The values were used to derive the beta rose
	dose rates	rates
Water content of sediment	20 ± 10 % wgt	15 ± 5 % wgt
Dose rate conversion factors	Guérin et al. (2011)	Adamiec and Aitken (1998)
In situ gamma measurements	No (gamma dose rate calculated from radioelement contents)	Yes (Nal probe connected to a Inspector1000 Canberra multichannel analyzer) on a section close to the excavation area.
		Throshold mothed (Marsian and Falguares
		2007) A mean gamma doce rate was derived
		from the 4 in situ measurements performed within layer D2.
Cosmic dose rate	Calculated from present-day depth (according with	Calculated using a three stage model evolution
	the tables of Prescott and Hutton, 1988, 1994)	based on Jamet's geological interpretation
		(according with the tables of Prescott and
		Hutton, 1988, 1994)
Age calculation program	DATA program (US-ESR and CS-USESR ages) (Grün, 2009)	USESR (US, Shao et al., 2014) and DATA (CS-US, Grün, 2009) programs

Analytical protocol	MNHN	BRGM
Preparation	Quartz extraction and purification proc	edure following Voinchet et al. (2004)
Selected grain size	100-20)0 μm
Number of irradiation steps	10)
D <u>max</u>	9,870 Gy	11,700 Gy
ESR signal	Al (Toyoda and Falguères, 2003)	Al (Toyoda and Falguères, 2003),
		Ti-Li and Ti-H (Toyoda et al., 2000 ;
		Tissoux et al., 2008)
ESR intensity	Al -From the top of the peak at g = 2.018 an	d the bottom of the 16th peak at g = 2.002
		Ti-Li - from the bottom of the peak at g =
		1.913 to the baseline
		Ti-H - from the bottom of the doublet at g
		= 1.915 to the baseline
Fitting function (data weighting)	E+L (1	L/I <u>2</u>)
Fitting program	Ori	gin
Alpha efficiency	0.15 ± 0.10 (Laur	ent et al., 1998)
Sediment radioelement contents	Determined	d by HRGS
Post Rn disequilibrium in the U-238 series	No disequilibri	um observed
Water content of sediment	Measured values (5-8 % wgt)	15 ± 5 % wgt (measured values :10-12%
		wgt)
In situ gamma measurements	Yes (Nal probe, Inspector 1000, Canberra),	Yes (Nal probe, Digidart LF gamma,
	directly at the sampling spot	Ortec), directly at the sampling spot
Cosmic dose rate	Calculated from present-day depth (accore	ding to the tables of Prescott and Hutton,
	1988, 1	1994)
Grain size attenuation for alpha and beta	Brennan (2003) and E	Brennan et al. <u>(</u> 1991)
dose rate		
Dose rate factor conversions	Adamiec and A	Aitken (1998)
Age calculation program	ESR MNHN	program

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		Stratigraph units	ical Lithostratigraphical descriptio (Lautridou, 1985 ; Jamet, 2014	n Biomarkers palaeoclimatic inference Ch J) (Lautridou, 1985 ; Jamet, 2015) Occh	ronological data (Stremme, 1985 ; ietti et al., 1987; Balescu et al., 199	Geolo 97) Guaquerel, 1984; Lautridou	gical 1,1985	interpretations 5 Jamet, 2014	
30	h A.	5 - X					MIS		MIS
			Flint gravels			Periglacial slope deposits	4-2	Slope deposits	4-2
28	¥	к	Sands with iron bands			Paleosol (Interglacial) on slope deposits	MIS 5	Paleosol	MIS 5
26 24 22			bedded sands and flint gravels	Cold and dry malacofauna (reworked ?)		Fluvio-aeolian unit (slope wash)		Fluvial to slope deposits (tidal input ?)	MIS 6
		<mark>е н</mark>	Humic sands	Temperate mammal fauna, but cold and dry malacofauna		Paleosol on fluvio-aeolian unit (climatic improvement)		Supratidal paleosol	
20		G F	Sands and flint gravels	Warm temperate mammal fauna (similar to D1) but cold malacofauna		Paleosol on solifluxion deposits (possibly interstadial)	MIS 6	Supratidal paleosol on slope deposits	
16		E	bedded sands	Rare more temperate microfauna and malacofauna Fig	jure 5 -	Fluvio-aeolian unit with paleosol (E2)		Tidal deposits (inner estuary)	MIS 7
14		De De	Clayey silt	Idem than D2		Paleosol (more cold than D2)		Mudflat deposits	
	7 5 5	D2	Humic silty sands	Cool mammal fauna and malacofauna		Paleosol (climatic degradation)		Supratidal paleosol	
12		D1	interbedded sands and silty clay	Fresh to brackish water Temperate mammal fauna foraminifera and ostracoda	≈ 200 (ESR shell) 198 ±26(TL, n=1) 196 ±23 (IRSL, n=3)	Fluvial deposits with marine influence (climatic optimum)	MIS 7	Tidal channel-flat deposits (inner estuary)	
8								Wandering fluvial deposits	
6			Gravels and sands	Mostly cold mammal fauna		Perglacial fluvial point-bar with head deposits (coombe-rock)	MIS 8	Braided fluvial deposits	MIS 8
2		B	interbedded sands and silty clay	Fresh to brackish water; temperate nalacofauna, foraminifera & ostracoda	313 ±33 (TL, n=1) 314 ±32 (IRSL, n=5)	Fluvial unit with estuarine influence	MIS 9	Tidal deposits (inner estuary)	MIS 9
0			Gravels and sands	-		Periglacial fluvial unit	MIS 10	Braided fluvial deposits	MIS 10
m	G.J 201	2							



Figure 3 - Tourville



Figure 4 - Tourville







		13 <u>02</u> HD+	TVL1301	TVL1:	8031			-
ESR samples 70		⊢ Tourvil Tourville Tourville Tourville	lle 4 2 3 2 1				◆ MN	HN AI
	o	TVL1304	⊦ ⊢		Tourville	6	BRG ⊃BRG OBRG	im ai Im ti-li Im ti-h
0 2	00 4	00 60	10 8	00 1	000 1	200 14	400 10	500 18

Figure 6 - Tourville

