A multi-technique dating study of two Lower Palaeolithic sites from the Cher valley 1 2 (Middle Loire Catchment, France): Lunery-la Terre-des-Sablons and Brinay-la Noira Mathieu Duval<sup>1,2</sup>, Pierre Voinchet<sup>3</sup>, Lee J. Arnold<sup>4</sup>, Josep M. Parés<sup>2</sup>, Walter Minnella<sup>5</sup>, 3 Verónica Guilarte<sup>6</sup>, Martina Demuro<sup>4</sup>, Christophe Falguères<sup>3</sup>, Jean-Jacques Bahain<sup>3</sup> & Jackie 4 Despriée<sup>3</sup> 5 6 <sup>1</sup>Australian Research Centre for Human Evolution (ARCHE), Environmental futures Research 7 University, NATHAN 8 Institute, Griffith QLD 4111, Australia. E-mail: 9 m.duval@griffith.edu.au <sup>2</sup> Geochronology and Geology, Centro Nacional de Investigación sobre la Evolución Humana, 10 Paseo de Atapuerca, 3, 09002 Burgos, Spain 11 <sup>3</sup> UMR 7194 HNHP, Museum National d'Histoire Naturelle, Département Homme et 12 environnement, 1 rue René Panhard, 75013 PARIS. 13 <sup>4</sup> School of Physical Sciences, Institute for Photonics and Advanced Sensing (IPAS) and 14 Environment Institute, University of Adelaide, North Terrace Campus, ADELAIDE, SA 5005, 15 16 Australia <sup>5</sup> PH3DRA Laboratories (PHysics for Dating Diagnostic Dosimetry Research and 17 Applications), Dipartimento di Fisica e Astronomia, Università di Catania & INFN – Catania, 18 Via Santa Sofia 64, 95123 Catania, Italy 19 <sup>6</sup> Departamento de Didáctica de las Ciencias Experimentales. Facultad de Educación y 20 21 Humanidades de Melilla. Universidad de Granada, Spain. 22 23 24 25

#### 26 Abstract

27 We present the results of a new dating study carried out at Lunery-la Terre-des-Sablons (LTS) and Brinay-la Noira (BN), two key Lower Palaeolithic sites located in deposits 28 29 associated to the Cher River (Middle Loire Catchment, France). These sites preserve abundant Mode 1 and Mode 2 lithic industries, and are considered as among the oldest evidence of 30 hominin presence in Western Europe north of the 45°N latitude. Following a multi-technique 31 approach combining electron spin resonance (ESR), single-grain thermally-transferred 32 optically stimulated luminescence (TT-OSL) dating of quartz grains and palaeomagnetism, we 33 obtain new chronological constraints for the sedimentary sequence, and the associated lithic 34 35 assemblages, at the two sites.

The new independent dating results derived from each method are consistent and in overall agreement with existing ESR and terrestrial cosmogenic nuclide (TCN) age estimates, except for the Lowermost Unit 3 at LTS. By integrating all of the previous and new dating results, we derive combined age estimates of 772-735 ka and  $665 \pm 29$  ka for the fluvial sands at LTS (Unit 1) and BN, respectively. These two distinct aggradation phases may tentatively be correlated to interglacial/glacial transitions associated with Marine Isotope Stage (MIS) 19-18 for the former and MIS 17-16 for the latter.

At BN, an age range of 638-676 ka may be proposed for the hominin occupation after
combining numerical age results and geological evidence. This result is consistent with the
initial chronology proposed by Moncel et al (2013) [Moncel, M.H., Despriée, J., Voinchet, P.,
Tissoux, H., Moreno, D., Bahain, J. J., Courcimault, G., Falgueres, C. (2013). Early evidence
of Acheulean settlement in north-western Europe – la Noira site, a 700 000 year-old occupation
in the Centre of France. Plos One, 8(11): 1-22] and confirms that BN is among the oldest
Acheulean occurrences in Western Europe.

At LTS, the mean age of  $710 \pm 50$  ka obtained for the sandy layer in Unit 3 provides a minimum age constraint for the archaeological level located below. The major sedimentary disconformity observed between the fluvial deposits and the underlying pebble layers hosting the lithic artefacts suggests that the true age of the artefacts might be significantly older, probably Early Pleistocene given their similarities with other Mode 1 assemblages identified in Western Europe. However, further refinement of the Mode 1 chronological inference at LTS remains difficult at this stage. Finally, these new dating results show the importance of using the Multiple Centre approach
for ESR dating of quartz grains, and confirm the value of combining different dating methods
in order to build more robust chronologies for Lower Palaeolithic sites in Europe.

From a methodological point of view, the dating results presented here are especially
encouraging for the reliability of the ESR method applied to optically bleached quartz grains.
This is one of the very first studies demonstrating that samples independently dated by two
different laboratories may produce generally reproducible ESR age results.

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### 66 Introduction

67 Over the last three decades, the Quaternary fluvial deposits of the Loire catchment and its tributaries (France) have been extensively and systematically studied from a multidisciplinary 68 69 perspective. This long-term investigation initiated by a team of researchers from the Muséum National d'Histoire Naturelle (MNHN) in Paris, France, has led to the identification of different 70 stepped terrace systems for the Loir, Cher and Creuse valleys (see an overview in Voinchet et 71 al., 2010 and Depriée et al., 2011). The deposits have been chronologically constrained by an 72 unprecedented large number of Electron Spin Resonance (ESR) ages (n>200) based on 73 74 optically bleached quartz grains. Most of the work has been focused on the measurement of the aluminium (Al) centre (e.g. Voinchet, 2002; Voinchet et al., 2010; Despriée et al., 2011; Garon 75 et al., 2017), although a few attempts at dating the titanium (Ti) centres have also been reported 76 (Tissoux et al., 2007). These dating results have established a robust and detailed regional 77 chronostratigraphic framework for the fluvial deposits covering most of the Pleistocene. 78 Additionally, a systematic archaeological survey of the fluvial terraces has led to the 79 80 identification of several tens of localities with Palaeolithic assemblages, some of which are amongst the oldest evidence of hominin presence in Western Europe (e.g. Despriée et al., 2006, 81 82 2011, Moncel et al., 2013). In particular, two localities are of major interest in the Cher valley (Middle Loire catchment): Lunery-la Terre-des-Sablons and Brinay-la Noira. The first of these 83 84 sites has provided Mode 1 (or Oldowan, depending on the terminology employed; Carbonell et al., 2015) lithic artefacts dated to between 0.9 and 1.1-1.2 million years ago (Ma) (Despriée 85 et al., 2017a), while the latter has yielded a Mode 2 (or Acheulean) assemblage dated to 600-86 87 700 ka (Moncel et al., 2013). The chronology of each site has been constrained by ESR ages 88 from several samples collected throughout the sequences. These results are internally consistent and stratigraphically coherent at a given site, producing an apparently robust 89 chronostratigraphic framework. That said, unequivocal independent age control is absent from 90 these sites. The only reported attempt so far has been made with terrestrial cosmogenic nuclide 91 (TCN) burial dating, which led to somewhat inconclusive results according to the authors (Shen 92 et al., 2012). For a long time, cross-comparisons have been almost impossible, as the geological 93 context and reported age of these sites has strongly limited the use of other dating methods. 94 95 For example, there is no suitable material for U-series or Ar-Ar dating applications. Moreover, the absence of fossils at these sites precludes not only any biochronological inference but also 96 the use of the ESR method applied to teeth. Additionally, the limited thickness of the 97 sedimentary sequences and the predominantly unconsolidated coarse sand deposits are not 98

ideally suited for a proper and detailed magnetostratigraphic study. Finally, the dose saturation
limitation of conventional optically stimulated luminescence (OSL) signals has typically
restricted the applicability of this sediment dating method to late Middle to Late Pleistocene
deposits, i.e. much younger than the published age of the two sites.

However, methodological advances over the past two decades have expanded the 103 104 applicability of dating techniques and improved their reliability. In particular, new extendedrange luminescence dating techniques such as single-grain thermally transferred OSL (TT-105 OSL) have provided reliable age estimates for Early Pleistocene deposits (e.g., Arnold et al., 106 2015; Bartz et al., 2018). Since the publication of the ESR ages at Lunery-la Terre-des-Sablons 107 and Brinay-la Noira, there have also been a number of significant methodological 108 developments in the ESR dating approach for optically bleached quartz grains. Among them, 109 the exponential+linear (EXP+LIN) function was recently found to be more appropriate than 110 the Single Saturating Exponential (SSE) function for fitting the ESR intensities of the Al centre 111 112 (Duval, 2012; Voinchet et al., 2013). The most significant recent breakthrough is undoubtedly 113 the increasing use of the Multiple Centre (MC) approach in ESR dating application studies. First proposed by Toyoda et al. (2000), it has progressively become a standard requirement in 114 115 the field of ESR dating of optically quartz grains (e.g. Duval et al., 2017a; Pereira et al., 2018). Based on the systematic measurement of both the Al and Ti centres in a given quartz sample, 116 117 it is considered as the best way to evaluate the inherent uncertainty associated with possible incomplete resetting of the Al signal prior to sediment deposition (see an overview in section 118 2 of Duval et al., 2017a). 119

In light of these recent advancements, a series of new ESR samples was collected in April 120 2011 at Brinay-la Noira and Lunery-la Terre-des-Sablons sites (Cher department) with the aim 121 of re-examining and refining the existing ESR chronologies of the sites. The new samples were 122 processed at the Centro Nacional de Investigación sobre la Evolución Humana (CENIEH) 123 following the standard MC procedure described in Duval et al. (2017), i.e. in a completely 124 independent way to those produced earlier by the MNHN research team for the same sites. 125 126 Additional samples were also collected for extended-range luminescence dating and 127 palaeomagnetic analyses in order to obtain independent age control and evaluate the accuracy of the ESR results. 128

In summary, the present multi-technique dating study aims not only at evaluating the reproducibility and accuracy of the ESR dating method applied to optically bleached quartz grains, but also at further constraining the chronology of two key Lower Palaeolithic localities located north of the 45°N latitude. The general implications of the refined chronologies will
also be discussed, and in particular regarding the stratigraphic interpretation of the sites.

Finally, because ESR, Luminescence and TCN age uncertainties are typically reported at a 1- $\sigma$  confidence level (e.g. Duller, 2008; Duval et al., 2017b; Carbonell et al., 2008; Arnold et al., 2015), this common practice will apply here as well: all numerical ages mentioned in the present manuscript will be given with their corresponding 1- $\sigma$  errors, unless mentioned otherwise.

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### 140 1. Study area

141 *1.1. The Cher valley* 

142 A total of nine stepped terraces have been identified and extensively dated by ESR in the Cher valley over ~50 m of vertical incision (Fig. 1). Named sequentially from A (current 143 144 floodplain deposits) to I (+45 m above the bedrock floor of A), these terraces have chronologies covering the last ~1.3 Ma (Voinchet et al., 2010). Archaeological surveys of those deposits has 145 led to the discovery of several Palaeolithic sites with lithic artefacts attributed to different 146 techno-complexes and found in primary position (i.e., not reworked; see a detailed overview 147 in Despriée et al., 2011). The oldest artefacts, characterised as Mode 1 assemblages, were found 148 in stacked terraces F (+28-34 m) and G (+34-40 m) at the same locality of Lunery-la-Terre-149 des-Sablons, and have been dated to about 0.9 and 1.1-1.2 Ma, respectively (see further detail 150 in section 1.2.). Mode 2 assemblages have been documented at Brinay-la Noira, Gièvres-la-151 Genetière and Gièvres-la-Plaine-de-la-Morandière localities (Loir-et-Cher department), all 152 correlated to fluvial terrace D (+14-21 m) and dated to about 650 ka (see Despriée et al., 2011 153 and references therein). Finally, a lithic assemblage with more advanced features (Levallois 154 155 debitage) has also been reported in Terrace B (+5-11 m) at Gièvres-la Morandière (Loir-et-Cher department), and dated to around 370 ka (Fig. 1). Together, these occurrences indicate 156 157 the recurrent presence of hominins in the area since the latest part of the Early Pleistocene.

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### Figure 1 approx. here

159 *1.2. Lunery-la Terre-des-sablons (LTS)* 

160 The LTS locality corresponds to a former sand quarry, a few km north of the village of 161 Lunery. Stratigraphic interpretation of the site based on sedimentological and chronological 162 evidence combined with field observations have led to the identification of three superimposed 163 sandy units (labelled 1 to 3 from top to bottom) deposited on the Oxfordian limestone bedrock, which has been identified at different depths across the site (Fig. 2). These units are preserved on a tilted main block (called "*les Sablons*" block) bordered on the western edge by the socalled Rosières fault and by the Cher river in the East. Successive fault reactivations until the Pleistocene have broken the Jurassic limestone in long and narrow smaller blocks. The course of the Cher river is presently flowing about 1 km east from the site (further details about the geological context may be found in Despriée et al., 2007, 2017a and Voinchet et al., 2007).

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### Figure 2 approx. here

Presently, the coarse red sandy Unit 3 is deposited on the lower block (-12m below the top 171 of the sequence; Fig.3), while the beige sandy Unit 2 is deposited on the middle block (-9 m), 172 173 both with a similar horizontal extension of about 50 m. The sandy Unit 1 is deposited on the highest limestone floor (-5m) over a 500-m width and covers both units 3 and 2 (Despriée et 174 al., 2017a; Fig. 2). The current relative altitude of Unit 1 is +29-34 m, but geological evidence 175 suggest that this altitude may have been somewhat higher in the past, i.e. before the les Sablons 176 177 block was downthrown, probably concomitant with the overstretching of the Cher graben (Despriée et al, 2017c). Similarly, the older Units 2 and 3 that are now covered by Unit 1 were 178 179 most likely originally deposited at this relative altitude or possibly even higher. The absence of others sedimentary remnants from these two units suggests that they were probably deposited 180 181 higher on the previously eroded western slope (see further details in Despriée et al., 2017a).

Unit 3 is about 2.20 m thick and lies on top of the Oxfordian limestone located about 12 m 182 below the surface. The bottom part of the lower layer (a) consists of ca. 30 cm of periglacial 183 diamicton containing blocks, cobbles and pebbles of materials derived from Neogene river 184 deposits from the plateau and the slope, and mixed with clayey sand and iron pisoliths. This 185 deposit is cryoturbated, with polygons and clusters of cobbles (Despriée et al., 2017a). A Mode 186 187 1 lithic assemblage (> 700 artefacts) divided into three archaeological horizons has been identified within the basal diamicton of layer a. The raw materials used are Jurassic oolitic 188 189 silicifications and lacustrine millstone slabs. There is no evidence of transport on the artefacts, suggesting that they were found in primary position (Despriée et al., 2010a and b). The 190 191 diamicton is overlain by a 20 cm-thick bed of locally derived pebbles and small cobbles with structures that suggest deposition by slope wash processes. Finally, layer b above is made of 192 193 1.70 m-thick cross-bedded sands. Full details about the stratigraphy and site formation processes may be found in Despriée et al., 2017a (in particular see Figure 27 of Despriée et al., 194 195 2017a).

196 The three sediment samples collected from the fluvial deposits of layer b have provided consistent ESR results (Al centre) of  $1102 \pm 119$ ,  $1174 \pm 122$  and  $1227 \pm 123$  ka (Despriée et 197 al., 2017a; Detailed numerical results may be found in Supplementary Material, Table S1). The 198 derived weighted mean age of  $1166 \pm 140$  ka may be interpreted as a minimum age constraint 199 200 for the lithic artefacts found stratigraphically below in layer *a*. These existing dating results mean that LTS preserves one of the oldest evidence of hominin presence in Europe, with a 201 202 chronology similar to that of Fuente Nueva 3 (Duval et al., 2012) and Atapuerca Sima del Elefante TE9 (Carbonell et al., 2008). Finally, <sup>26</sup>Al/<sup>10</sup>Be burial dating has also been performed 203 on one quartz sand sample from layer b of Unit 3, about 11 m below the surface (Shen et al., 204 2012) and at the same stratigraphic position as the MNHN ESR samples. Assuming post-burial 205 nuclide production or the absence of post-burial nuclide production resulted in very close 206 estimates of  $750 \pm 240$  and  $710 \pm 210$  ka, respectively, which are younger than the 207 corresponding ESR ages. The large uncertainties associated with the TCN age estimates arise 208 from the high natural Al content of the sediments (>100 ppm), which led the authors to consider 209 their results as preliminary. 210

Unit 2 is preserved over a maximum thickness of about 4.5m and comprises two coarse sand layers (*a* and *b*) (Fig. 3) positioned on top of the Oxfordian limestone located about 10 m deep below surface. Mode 1 lithic artefacts (mostly cores and choppers) were found at the base of layer *a* (Despriée et al., 2010, 2011). ESR dating of two samples (Al centre) from the middle of layer *b* have provided consistent age results of  $936 \pm 49$  ka and  $949 \pm 94$  ka (Despriée et al., 2017a; Table S1). The derived weighted mean age of  $939 \pm 85$  ka may be interpreted as a minimum age constraint for the lithic artefacts of Unit 2 (Fig. 3).

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### Figure 3 approx. here

Finally, Unit 1 has a thickness of 5.5 m and comprises two reddish layers (*a* and *b*) of indurated coarse sands in a clay matrix (Fig. 3). Layer *a* lies on top of clays derived from the weathering of the limestone bedrock. ESR dating yielded two consistent results of  $808 \pm 71$ and  $839 \pm 79$  ka for layers *a* and *b*, respectively (Despriée et al, 2017a; Table S1). A third sample collected from the same unit but at another section located a few hundred meters from the site has provided a very similar result ( $831 \pm 103$  ka).

In summary, the sand layers of the three units identified at different relative altitudes show different sedimentary facies and also yield different depositional ages. This evidence suggests that the LTS Units are not coeval, but rather correspond to different aggradation events. Based on the existing dating results, Units 1, 2 and 3, have been tentatively correlated to fluvial terraces E, F and G of the general chronostratigraphic framework of the Cher Valley, respectively, while more recent tectonic events have significantly affected the original geometry and morphology of the deposits (Despriée et al., 2007, 2011, 2017a and b).

#### 232 *1.3.Brinay-la Noira (BN)*

The 6-m thick sedimentary sequence at BN is correlated with fluvial terrace D (+14-21 m) 233 of the Cher valley. It lies on top of an Upper Eocene-Oligocene lacustrine limestone and 234 greenish clay (so-called "Calcaires et Argiles lacustres du Berry" formation). Four different 235 layers have been locally identified above the bedrock, named a to d from bottom to top 236 237 (Despriée, et al., 2017b; Fig. 4). Layer a, whose thickness laterally varies between 30-90 cm, is made of a very coarse sandy and clayey deposit, including numerous endogeneous pebbles, 238 239 jurassic silicifications and millstone slabs in the lower part of the layer (sub-layer al). The upper part (sub-layer a2) is interpreted as slope deposits. Above, layer b is composed of about 240 241 5 m of fluvial coarse sands that get finer to the top, and is covered by layer c, which comprises fine and middle-sized gravel deposits with many iron pisoliths, millstone slabs and well-242 243 rounded quartz pebbles. On top, layer d is a sandy brown silt layer including millstone debris with orange patina that may locally reach a thickness of 70 cm. 244

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### Figure 4 approx. here

Lithic artefacts (n = 630) were found *in situ* within layer *a1* during fieldwork and excavations performed between 2003 and 2014. The assemblage is dominated by large cutting tools, cores and flakes, and crude core-tools, mostly made from millstone slabs (90%), which overall conforms well with other early Acheulean sites in Europe (Moncel et al., 2013, 2018). Stratigraphic and sedimentological evidence suggest that the upper part of the slope deposits (layer a2) is somewhat younger than the lithic assemblage and accumulation of blocks and stones at the base of layer a (Despriée et al., 2011).

Six ESR sediment samples have been dated from the fluvial sands of layer *b*. The measurement of the Al centre provided consistent ages between 600-700 ka (Moncel et al., 2013; Table S2). Consequently, a weighted mean age of  $665 \pm 55$  ka was derived for this unit, which may be interpreted as a minimum estimate for the underlying lithic assemblage located within layer *a*. These results make Brinay-la Noira one of the oldest Acheulean site in Europe (Moncel et al., 2018). 259 Three additional, unpublished, ESR dating results derived from the same analytical procedure as in Moncel et al. (2013) (see details in Table S2) provide additional chronological 260 constraints at the top and base of the sedimentary sequence. One sample collected from layer 261 c gave a maximum age of  $448 \pm 46$  ka for the upper part of the sequence (note that this age has 262 been briefly mentioned in Iovita et al., 2017, but no further details were provided). Although 263 this age is stratigraphically consistent with the others from layer b located below, it is 264 nevertheless suspected to be underestimated on stratigraphic grounds. The identification in the 265 stratigraphic sequence of a continuous and horizontal bed of gravel at the base of layer c266 intersected by several ice wedges (Despriée et al, 2017b) indicates the presence of a 267 palaeosurface probably created by erosion and short movements of sediments on the slope. 268 Runoff waters have most likely removed most of the fine alluvium and concentrated the gravel. 269 Consequently, the sands extracted from this level and dated by ESR were most likely, at least 270 in part, reworked and exposed to sunlight during this erosion process, inducing some resetting 271 of the signals. Two additional samples extracted from the upper (a2) and lower (a1) parts of 272 layer a have provided ages of  $645 \pm 30$  and  $1090 \pm 61$  ka, respectively. The first of these ages 273 is very close to those obtained from layer b, whereas the second age is significantly older than 274 the published ESR dataset for layer b. This age discrepancy could be due to either incomplete 275 276 resetting of the Al signal (as suggested by the different bleaching coefficients measured; Table S2) or contamination of the quartz samples by another population of older grains. Indeed, the 277 278 lowermost sample was collected from slope deposits next to the accumulation of millstone plates and blocks. This very poorly sorted heterometric sand and gravel sediment most likely 279 280 experienced little or even no river transport, which did not lead to a complete resetting of the 281 ESR signal in quartz grains.

Finally,  ${}^{26}$ Al/ ${}^{10}$ Be burial dating was performed on one quartz sand sample from layer *a* (Shen et al., 2012). Assuming either post-burial nuclide production or the absence of post-burial nuclide production resulted in very close estimates of 730 ± 210 ka and 700 ± 200 ka, respectively. Again, this sample exhibits large associated dating uncertainties due to the same reason detailed earlier for LTS. These preliminary TCN results are consistent with the ESR chronology available at this site.

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289 2. Sampling

290 Bulk sediment samples were collected by hammering a thick PVC tube into the section in order to avoid exposure to sunlight. Two ESR samples were collected at LTS (Fig. S1): one 291 (LUN1103) from the upper Unit 1 and another (LUN1101) at the bottom of the sequence within 292 Unit 3. Unit 2 was not sampled due to the absence of accessible outcrop in 2011. Two samples 293 294 were also taken at BN (Fig. S2), one (NOI1101) at the top of layer b and the second (NOI1104) from within layer a at the bottom of the local sequence. In situ measurement of the gamma 295 dose rate was systematically performed for each sample at the exact same sampling spot with 296 a NaI probe connected to an Inspector-1000 multichannel analyser. Additional raw sediment 297 298 samples were also collected for laboratory analyses.

In parallel, samples were collected for single-grain TT-OSL dating and palaeomagnetic analyses at the same two localities. TT-OSL sample collection and *in situ* dosimetry assessments followed the procedures outlined in Demuro et al (2015). At LTS, TT-OSL sample CRF11-2 was collected within Unit 3, ~8 m to the left of LUN1101 (Fig. S1 C). At BN, TT-OSL sample CRF11-4 was taken from layer *a*, ~40 cm to the left of ESR sample NOI1104 (Fig. S2).

305 Sampling for palaeomagnetic analyses was challenging at the two sites: the deposits are dominated by unconsolidated coarse-grained sands, which precludes a proper high-resolution 306 307 magnetostratigraphic study of the sequences. Whenever possible, fine-grained sandstones or 308 siltstone layers were selected for sampling. These samples were obtained by cutting small cubes from oriented blocks (ca 300 cc) using a small ceramic knife. Because of their friability, 309 block samples were often impregnated in the field with a 1:1 sodium silicate solution for 310 magnetic analysis. At LTS, we sampled two locations within Units 1 and 3 (LUN1 and LUN2; 311 Fig. 3 & Fig. S1), from which several subsamples were collected and analysed in the laboratory. 312 At BN, we sampled three different horizons (Fig. 4). Samples NOR1-1 and NOR1-2 came from 313 the archaeological layer and consist of clayish sandstones. Samples NOR1-3, 4 and 5 were 314 collected from within the upper part of layer *a*. Lastly, sampling site NOR1-6 is located slightly 315 316 north of the previous site and corresponds to a thin (4 cm) clayish layer within b.

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### 318 **3. Methods**

319 *3.1. ESR dating* 

Sediment samples were prepared in the laboratory under conditions of limited illumination
 following the standard procedure at CENIEH (e.g. Duval et al., 2017a). The 100-200 μm size

fraction was collected after wet sieving. HCl (36%) was used to dissolve carbonates and H<sub>2</sub>O<sub>2</sub> (30%) to eliminate organic matter. Heavy minerals and feldspars were removed with Sodium Polytungstate solutions having a density of rho ( $\rho$ ) =2.72 and 2.62 g/ml, respectively. Then, magnetic minerals were eliminated using Neodymium magnets. The resulting samples were treated with HF (40%) for 40 minutes to eliminate the remaining feldspars and to etch quartz grains. Finally, HCl (18%) was added in order to remove any soluble fluoride.

Quartz grains were dated by using the standard Multiple Aliquots Additive Dose (MAAD) method. Each natural sample was divided into 14 multi-grain aliquots. Twelve aliquots for each sample were irradiated using a <sup>137</sup>Cs Gammacell-1000 source (dose rate = 7.3 Gy/min, relative 1 $\sigma$ -uncertainty = 2.3%) to the following doses: 199, 398, 597, 995, 1492, 2487, 4974, 7959, 11938, 19896, 29844 and 49640 Gy. One aliquot was kept unirradiated (natural aliquot), while the last aliquot was exposed to a SOL2 (Dr Hönle) solar light simulator for about 1240 h, in order to evaluate the non-bleachable residual ESR signals of the Al centre.

335 ESR measurements were carried out at CENIEH (Burgos, Spain), with an EMXmicro 6/1Bruker X-band ESR spectrometer coupled to a standard rectangular ER 4102ST cavity. To 336 337 ensure constant experimental conditions over time, the temperature of the water circulating in the magnet is controlled and stabilized at 18 °C by a water-cooled Thermo Scientific NESLAB 338 ThermoFlex 3500 chiller, and the temperature of the room is kept constant at 20 °C by an air 339 340 conditioning unit. ESR measurements were performed at low temperature (~90 K) using a ER4141VT Digital Temperature control system based on liquid nitrogen cooling. Further 341 details about the setup and about its stability over time can be found in Duval and Guilarte 342 Moreno (2012). 343

In accordance with the Multiple Centre method defined by Toyoda et al. (2000), the ESR 344 345 signals of both the Al and Ti centres were measured. For the Al centre, the following acquisition parameters were used: 10 mW microwave power, 1024 points resolution, 20 mT sweep width, 346 347 100 kHz modulation frequency, 0.1 mT modulation amplitude, 40 ms conversion time, 10 ms time constant and 1 scan. In contrast, the ESR signal associated with Ti centres was measured 348 349 as follows: 5 mW microwave power, 1024 points resolution, 20 mT sweep width, 100 kHz 350 modulation frequency, 0.1 mT modulation amplitude, 60 ms conversion time, 10 ms time 351 constant and 2 to 4 scans (depending on the aliquot). Each of the 14 aliquots of a given sample were measured 3 times after a  $\sim 120^{\circ}$  rotation in the cavity for both Al and Ti signals in order 352 353 to consider angular dependence of the signal due to sample heterogeneity. All measurements were repeated three times over distinct days in order to check the repeatability of the D<sub>E</sub> values 354

355 (Tables 1 & 2). Consequently, three sets of 14 data points were obtained for each signal
356 measured (Ti and Al) in a given quartz sample.

The ESR intensity of the Al signal was extracted from peak-to-peak amplitude measurements between the top of the first peak (g=2.0185) and the bottom of the 16th peak (g=1.9928) (Toyoda and Falguères, 2003). The ESR intensity of the Ti centres was measured as in Duval and Guilarte (2015): peak-to-peak amplitude between g=1.979 and g=1.913 (option A), peak-to-baseline amplitude around g=1.915 (Ti-H, Option C), peak-to-baseline amplitude around g=1.913-1.915 (option D), and peak-to-baseline amplitude around g=1.979 (option E).

For each aliquot, ESR intensities of Al and Ti centres were corrected by the corresponding 363 364 receiver gain value, number of scans, mass and a temperature correction factor (Duval and Guilarte Moreno, 2012). The fitting procedures were carried out with the Microcal OriginPro 365 9.5 software using a Levenberg-Marquardt algorithm by chi-square minimisation. For the Al 366 centre, an exponential+linear function (EXP+LIN) was fitted through the data points (see 367 368 equation in Duval et al., 2017a), and data were weighted by the inverse of the squared ESR intensity ( $1/I^2$ ). D<sub>E</sub> values were obtained by extrapolating the EXP+LIN function to the residual 369 370 ESR intensity (Table 1), as in the Total Bleach method described in Forman et al. (2000). This approach is based on the assumption that the signal of the Al centre has been fully reset to its 371 residual level during sediment transport (Voinchet et al., 2003 & 2004). If not, then the dose 372 373 result derived from the Al centre should be interpreted as a maximum possible estimate of the true burial dose. For the Ti centre, we have used the function labelled as Ti-2 in Duval and 374 Guilarte (2015), in order to describe the non-monotonic dose dependence of the ESR signal at 375 high doses. Data were weighted by the inverse of the squared experimental error  $(1/s^2)$  and D<sub>E</sub> 376 values were obtained by back extrapolation to Y=0 (Table 2). For each sample, final dose 377 response curves (DRCs) were obtained by pooling all the repeated measurements in a single 378 plot (Supplementary material, Fig. S3 and S4), as recommended by Duval (2012). The final  $1\sigma$ 379 D<sub>E</sub> error is derived from the combination of the errors on the fitting and on the calibration of 380 the irradiation source (2.3 %). 381

The total dose rate value was derived from a combination of *in situ* and laboratory measurements. External gamma dose rates were derived from *in situ* measurements by using the "threshold technique" (Duval and Arnold, 2013). For each dated sample, the corresponding radioelement (U, Th, K) concentrations in the raw sediment were determined by ICP-MS analysis of about 5 g of dry material (Supplementary material, Table S3). Concentration values were used to derive external alpha and beta dose rate components using the dose rate 388 conversion factors from Guérin et al. (2011). Potential disequilibrium in the U-238 decay chain was not directly evaluated. Nevertheless, the consistency between the gamma dose rate values 389 derived from *in situ* and laboratory measurements may be used as a good proxy to suggest the 390 negligible impact of such disequilibrium, if present. Dose rate values were calculated assuming 391 392 a mean grain size of 150 µm, and an assumed thickness removed by HF etching of 20 µm. Internal dose rate was assumed to be  $50 \pm 30$  uGy/a as in Duval et al., (2017). Values were 393 corrected with beta and alpha attenuation values for spherical grains (Brennan et al., 1991; 394 Brennan, 2003) and water attenuation formulae from Grün (1994). Actual water contents were 395 evaluated in the laboratory by drying the sediment at 50 °C in an oven during three weeks. 396 Results vary within a relatively narrow range from 7 to 12 % (wet weight) among the samples. 397 These values most likely underestimate the long-term water content at these sites, because the 398 samples were collected at shallow depths from the section surface (<30 cm). Some drying out 399 of the sediment profiles may have occurred prior to sampling because these sections had been 400 exposed for several years in the excavation area. For these reasons, dose rate evaluation was 401 performed with higher assumed values of  $20 \pm 5\%$ , which is consistent with the 21-22% (% of 402 dry sediment weight) used for the TT-OSL dating comparisons at BN and LTS. The large 403 associated error (5% at a  $1\sigma$  confidence level) covers a range from 10 to 30% at  $2\sigma$ , most likely 404 405 encompassing any significant long-term variability of the water content. The cosmic dose rate was calculated using formulae from Prescott and Hutton (1994), with depth, altitude and 406 407 latitude corrections (Prescott and Hutton, 1988).

ESR age calculation were performed using ESR-Qz, a non-commercial SCILAB based software, with error calculations based on Monte Carlo simulations, considering the following sources of uncertainties: concentrations, depth, water content, gamma dose rate, beta dose attenuation,  $D_E$  values. Test comparisons performed with DRAC (Durcan et al., 2015) show that both programs provide very close results for a given data set (Kreutzer et al., 2018). ESR ages are given with 1 $\sigma$  error ranges (Table 3).

414 *3.2. Extended-range luminescence dating* 

The comparative extended-range luminescence dating study at BN and LTS focuses on single-grain TT-OSL dating of quartz, following the successful application of this approach at several independently dated Lower Palaeolithic sites from southwest Europe (e.g., Demuro et al., 2014; Arnold et al., 2013, 2014; Arnold and Demuro, 2015). Single-grain TT-OSL dating was performed at the CENIEH Luminescence Dating Laboratory, Burgos, Spain, using the 420 instrumentation, methodological procedures and TT-OSL quality assurance criteria outlined in421 Arnold et al. (2014).

TT-OSL equivalent dose ( $D_E$ ) values were determined using the single-aliquot regenerativedose (SAR) protocol shown in Table S4, which has been modified to enable measurement of individual quartz grains. The suitability of the chosen  $D_E$  determination procedures was assessed by undertaking a 2000 Gy dose-recovery test on sample CRF11-4, following the approach outlined in Bartz et al. (2019). A net (i.e., natural-subtracted) measured-to-given dose ratio of 1.00 ± 0.06 was obtained for this sample, confirming the suitability of the SAR procedure under controlled laboratory conditions.

429 'Pseudo' single-grain TT-OSL D<sub>E</sub> measurements have been made by loading 125-180 um quartz grains into standard single-grain aluminium discs drilled with a 10×10 array of 300 µm-430 deep depressions (holes). It is estimated that ~7 grains are placed in each hole when using this 431 configuration (Arnold et al 2012a), though we are reasonably confident that true single-grain 432 resolution has been maintained in this study owing to the low frequency of grain-hole positions 433 that produce TT-OSL signals (62-67% of measured grain-hole positions did not produce any 434 statistically distinguishable TT-OSL T<sub>n</sub> signals for samples CRF11-2 and CRF11-4; Table S5). 435 Individual D<sub>E</sub> values were obtained from the first 0.17 s of laser stimulation with a background 436 subtraction derived from the last 0.25 s of stimulation. 437

Following the findings of Bartz et al. (2019), we have used an additional Fast Ratio (FR) 438 439 (Durcan and Duller, 2011) quality assurance criteria to ensure that potentially unsuitable grains displaying very slowly bleaching TT-OSL signals are not included in our final age assessments. 440 441 The FR has been calculated by comparing the counts in the initial channel (0.017 s) of the TT-442 OSL decay curve  $(L_1)$  with those in the middle part of the decay (average counts over 1.0–1.2) 443 s; L<sub>2</sub>) after subtracting a late light background count from the last 0.068 s (L3), according to 444 the equation  $(L_1-L_3)/(L_2-L_3)$ . Sensitivity tests involving assessments of increasingly stringent FR threshold versus weighted mean D<sub>E</sub> (e.g., Bartz et al., 2019) revealed that a FR acceptance 445 threshold of  $\geq 20$  was suitable to eliminate any potentially biasing effects associated with slow 446 decaying TT-OSL signals for these particular samples (Arnold et al., in prep). 447

Environmental dose rates have been calculated using a combination of *in situ* field gammaray spectrometry (e.g., Arnold et al., 2012b) and low-level beta counting (Bøtter-Jensen and Mejdahl, 1988), taking into account cosmic-ray contributions (Prescott and Hutton, 1994), an assumed minor internal alpha dose rate (Bowler et al., 2003), beta-dose attenuation (Mejdahl, 452 1979; Brennan, 2003) and long-term water content (Aitken, 1998). Further details of the dose
453 rate determination procedures are provided in Table 4.

### 454 *3.3. Palaeomagnetism*

455 All magnetic measurements were carried out in the Paleomagnetism Laboratory at the CENIEH. Analyses included measurement of the magnetic remanence in a shielded, three-axis 456 superconducting 755R-4K rock-magnetometer (2G Enterprises). Samples were demagnetized 457 using standard procedures including both thermal and alternating field demagnetization, based 458 on previous results from similar lithologies (Parés et al., 2018). Additional rock-magnetic 459 analyses include isothermal remanent magnetization, and hysteresis cycles, carried out to 460 461 determine the magnetic mineralogy that is responsible for the paleomagnetic signal. Characteristic Remanent Magnetization (ChRM) component directions were calculated for all 462 463 specimens using Principal Component Analysis, guided by visual inspection of orthogonal ("Zijderveld type") demagnetization plots (Zijderveld, 1967). Mean directions and associated 464 statistical parameters were estimated using Fisher's method (Fisher, 1953). Corresponding 465 Virtual Geomagnetic Pole Latitude was used to determine the magnetic polarity at each 466 sampling horizon (Supplementary Material Fig. S8). 467

468

### 469 **4. Results**

470 *4.1. ESR dating* 

- 471 4.1.1.  $\underline{D}_{\underline{E}}$  determination
- 472 4.1.1.1. Al centre

ESR data derived from the measurement of the Al centre are displayed in Table 1. Bleaching coefficients values vary between 50% and 60%, suggesting somewhat similar bleaching conditions for all samples. As a comparison, these values are close to those obtained from other samples located within the Iberian Peninsula, such as those from Cuesta de la Bajada or the Alcanadre fluvial terraces (55-60%, Duval et al., 2015; Duval et al., 2017a).

The quality of the ESR data collected is within acceptable standards when compared with previous studies. Measurement repeatability for a given sample is <4 %, except for sample LUN1103 (7.4%). This results in a higher D<sub>E</sub> variability of 13% over repeated measurements, while it remains <10% for the other samples. Goodness-of-fit is excellent for 3/4 samples with adjusted  $r^2 > 0.99$ . The remaining sample (NOI1104) has a slightly lower adjusted  $r^2$  value as a result of some scattered points in the lowest part of the curve (Fig. S3). In summary, there is a series of consistent evidence (measurement and D<sub>E</sub> variability, high goodness-of-fit) indicating the overall quality and reliability of the ESR dataset collected for these four samples.

487 4.1.1.2.Ti centres

The quality of the ESR data collected for the Ti-Li centre (option D) is good overall. Measurement repeatability achieved for the four samples is <1.5 % (Table 2). The variability of the D<sub>E</sub> values is good (<10%) for 2 of the 4 samples, while the remaining two samples display higher values of around 15-16%. Goodness-of-fit is generally relatively good (Fig. S4), with 3 samples displaying adjusted r<sup>2</sup> values between 0.98 and 0.99 and the last one >0.99.

Option A yields higher  $D_E$  estimates for 3/4 samples, with the exception being NOI1101 493 494 (Table 2). This pattern is consistent with previous observations by Duval and Guilarte (2015), and is due to the influence of the peak at g=1.979 (option E). The latter provides systematically 495 496 higher D<sub>E</sub> estimates (between +33% and 48% higher than option D). Previous studies have shown the potential of Option D for providing accurate age estimates in agreement with 497 independent age control (e.g. Duval et al., 2017a; Bartz et al., 2018; Mendez-Quintas et al., 498 2018). Consequently, the final ESR ages were derived from Option D in the present study. The 499 ESR fitting results derived from the other options are nevertheless made available in Table 2 500 so that the corresponding ages can be calculated and referred to in future studies if required. 501

502 Finally, it should be mentioned that D<sub>E</sub> estimates were also obtained from Option C (Ti-H) for comparison, but discarded for age calculation. Goodness-of-fit was found to be lower than 503 for Options A and D (0.96 < adj. r2 < 0.99), and the resulting D<sub>E</sub> estimates were found to be 504 between 40% and 75% lower than those obtained with Option D. Although the Ti-H centre has 505 506 been recently found to show great potential to date late Middle to Late Pleistocene deposits (e.g. Kreutzer et al., 2018; Mendez-Quintas et al., in prep.), it is still unclear whether it can be 507 508 reliable for older samples, and in particular for  $D_E$  values > 1000 Gy (Duval and Guilarte, 2015), as is the case in the present study. 509

510 4.1.1.3.The MC approach

The Al centre provides systematically higher  $D_E$  estimates (between +4 and 32 %) compared with Ti-Li centre Option D, although one may reasonably consider that NOI1101 yields Al results within error of its corresponding Ti-Li results (Tables 1 and 2). Following the principles of the MC approach, this pattern indicates that the ESR intensity of the Al centre was not fully reset during sediment transportation (with the exception of sample NOI1101), as the result of its slower bleaching rate when compared with that of the Ti centres (see Fig. 1 of Duval et al.,2017a).

### 518 4.1.2. ESR age estimates

The two samples from LTS provide ESR ages within error for a given centre: ~850-900 ka for the Al centre and ~650-730 ka for the Ti-Li centre (Table 3). These results suggest that Units 1 and 3 may be considered as almost coeval. In comparison, the ESR-Al ages obtained at BN provide a maximum possible chronology for the deposits ranging from ~680 to ~940 ka from top to bottom. In contrast, the Ti centre yields somewhat younger ages of ~650 to ~740 ka that are within error of each other, suggesting a rapid sedimentation for the different layers.

In accordance with the MC approach, the results derived from the Al centre are interpreted as maximum age constraints for the samples, while the Ti-Li centre is considered to provide a more accurate estimate of the burial age of the deposits.

528 4.2. Single-grain TT-OSL dating

The single-grain TT-OSL dating results are summarised in Table 4. Single-grain TT-OSL 529 530 measurements were made on 1900-2000 quartz grains per sample, with ~10% of these 531 individually measured grains deemed suitable for D<sub>E</sub> determination after applying the singlegrain quality assurance criteria of Arnold et al. (2014) and a FR acceptance threshold of  $\geq 20$ 532 533 (Table S5). Representative decay and dose-response curves for two accepted grains are shown in Supplementary Material Fig. S5. The single-grain TT-OSL dose-response curves for samples 534 535 CRF11-2 and CRF11-4 are all well-represented by a single saturating exponential function, and exhibit continued signal growth over high dose ranges of several kGy. 536

The single-grain TT-OSL D<sub>E</sub> distributions of CRF11-2 from LTS Unit 3 and CRF11-4 from 537 BN layer *a* display limited scatter and contain a single dose population, with the vast majority 538 539 of individual D<sub>E</sub> values being well represented by the weighted D<sub>E</sub> value (falling within the shaded band in the radial plots) (Supplementary Material Fig. S6). Neither of these samples are 540 considered to be significantly positively skewed according to the criterion outlined by Arnold 541 and Roberts (2011). Low to moderate overdispersion values of  $29 \pm 4\%$  and  $34 \pm 5\%$  were 542 obtained for samples CRF11-2 and CRF11-4, respectively, which are in agreement at  $2\sigma$  with 543 values reported elsewhere for well-bleached and unmixed single-grain TT-OSL samples (e.g., 544  $21 \pm 2\%$ ; Arnold et al., 2019). These D<sub>E</sub> distribution characteristics suggest that the LTS and 545 BN TT-OSL samples were not significantly affected by partial bleaching or any major post-546 depositional complications thereafter (e.g., sediment mixing or beta dose rate heterogeneity). 547

548 Consequently, representative single-grain TT-OSL burial dose estimates have been calculated549 using the central age model (CAM) of Galbraith et al. (1999).

### 550 *4.3.Palaeomagnetism*

Initial Natural Remanent Magnetization (NRM) intensities were on the order of 10<sup>-3</sup> to 10<sup>-</sup> 551 <sup>4</sup> A/m, which is well-above the noise level of the cryogenic magnetometer. Both thermal and 552 AF demagnetization were used to isolate the Characteristic Remanent Magnetization (ChRM), 553 although the former was proven to be more efficient at isolating the ChRM directions. 554 Demagnetization plots (Supplementary Material, fig. S7) reveal a secondary, soft 555 magnetization component in many samples. Such viscous magnetization is typically erased by 556 heating to 200-300°C. After removal of this secondary component, a ChRM direction was 557 defined. Most samples produced stable, well-defined ChRM directions, with maximum angular 558 deviation below 15° (see Table 5). Only normal polarity directions (north seeking and 559 downwards) were obtained using either method (thermal or alternating field), giving 560 confidence of the effectiveness of the cleaning procedure (Supplementary Material, fig. S8). 561 Specimens from sampling site LUN1 are an exception. Only two specimens produced 562 interpretable demagnetization paths, albeit they are somewhat noisy. Unfortunately, the lack of 563 enough vectors hampers computing a reliable linear regression for these specimens from 564 LUN1. It is noteworthy that after a strong overprint (up to 300-400 °C), some vectors above 565 450 °C suggest the presence of a higher stability component in the south quadrant, perhaps 566 revelaing a south-directed component strongly masked by the normal magnetic field. We have 567 not calculated ChRM directions for these two samples, which will not be further considered. 568 569 Samples from NOR1-6 sampling site did not produced any stable directions and therefore are not included in the discussion. 570

571 Maximum unblocking temperatures and coercivity spectra suggest that (low-Ti) magnetite 572 is the main mineral responsible for the stable magnetization, with some contribution of 573 hematite.

574

### 575 **5. Discussion**

- 576 5.1. ESR age comparisons
- 577 5.1.1. <u>Al centre</u>

578 In general, the ESR-Al results obtained for CENIEH and MNHN samples at LTS are in 579 agreement at  $2\sigma$  and indicate an Early Pleistocene chronology for the deposits of Units 1 and 3 580 (Fig. 5). However, closer evaluation of the two ESR-Al datasets reveals a noticeable age difference. The CENIEH ESR sample LUN1103 from Unit 1 provides an age estimate of 893 581  $\pm$  87 ka, which is in good agreement with the two MNHN ESR results available (black circles 582 in Fig. 5B:  $808 \pm 71$  and  $839 \pm 79$  ka; Table S1). In contrast, the ESR-Al age of LUN1101 (866) 583  $\pm$  100 ka) from the bottom of the local sequence is > 200 ka younger than the three closely 584 associated MNHN ages from Unit 3 (black circles in Fig. 5A:  $1102 \pm 119$ ,  $1174 \pm 122$  and 1227585  $\pm$  123 ka; Table S1). This age difference is most likely linked to the D<sub>E</sub> evaluation, and 586 especially to the fitting function used. This hypothesis is confirmed by the new D<sub>E</sub> evaluations 587 588 performed using the EXP+LIN fitting function for the MNHN samples (red circles in Fig. 5A and B; numerical values are given in Table S1) as recommended by Duval (2012): while 589 samples from Units 1 and 2 are largely insensitive to the use of this fitting function ( $\pm 1\%$  and 590 -4%, respectively; Table S1) those from Unit 3 yield significantly younger ages (by 100-230 591 ka, or -8 to -26 %; Table S1). The resulting ages of the Unit 3 samples range from 900 to 1100 592 ka when using the EXP+LIN fitting function (red circles in Fig. 5A). These results are, within 593 error, in good agreement with the CENIEH ESR-Al age estimate of  $866 \pm 100$  ka obtained for 594 sample LUN1101, which is also based on the use of an EXP+LIN fitting function. The 595 reduction of the D<sub>E</sub> value induced by the use of the EXP+LIN function is consistent with the 596 597 results obtained by Duval (2012) from a previous comparison study based on 15 quartz samples. In that study, the D<sub>E</sub> values derived from the EXP+LIN function were on average 33% 598 599 lower than those obtained from the SSE function. The magnitude of this systematic difference was nevertheless sample-dependent (ranging from -64 to -8% depending on the sample 600 601 considered), which may explain why some MNHN samples are significantly less affected by the choice of the fitting function. 602

603

## Figure 5 approx. here

At the BN locality, CENIEH sample NOI1101 from the top of layer b provides an Al age 604 of  $682 \pm 68$  ka, which is not only consistent with the nearby Cher2004-11 and Cher2004-12 605 606 sample ages ( $699 \pm 76$  and  $680 \pm 77$  ka; Table S2 & Fig. 4) but also with all the MNHN ESR 607 age estimates obtained for that layer (red circles in Fig. 5D). In contrast, the age of  $940 \pm 110$ 608 ka obtained for CENIEH sample NOI1104 at the base of the sequence is significantly older than that obtained for the stratigraphically associated MNHN ESR samples from the upper part 609 of layer a (a2) (red circle in Fig. 5C:  $645 \pm 30$  ka). This apparent age discrepancy most likely 610 originates from the D<sub>E</sub> values: these differ significantly by ~40% (1875 $\pm$ 87 Gy vs. 2639 $\pm$ 263 611 Gy), while both samples display similar dose rate values (2907±40  $\mu$ Gy/a vs. 2807±221 612

613  $\mu$ Gy/a). Interestingly, the bleaching coefficient of 57% measured for NOI1104 (Table 1) is much higher than that obtained for the MNHN sample (42%; Table S1). This suggest different 614 bleaching conditions for the two samples and different levels of ESR signal resetting was 615 achieved for the Al centre during sediment transport. Consequently, we consider that the 616 apparent age discrepancy is most likely due to lateral variations of the slope deposits at BN 617 (see Fig. 6 and 8 from Despriée et al., 2017b), whose complexity cannot be easily captured in 618 such generalised or composite stratigraphic logs. The MNHN and CENIEH samples were not 619 collected at the exact same spot within each layer, but were laterally distant by a few meters. 620 621 Consequently, the dose scatter (and the resulting age discrepancy) may simply be the result of the heterogeneity of the sandy matrix within slope deposits that may not have locally 622 experienced enough transport and/or sunlight exposure to fully reset the ESR-Al signal of 623 sample NOI1104. 624

In summary, the CENIEH and MNHN samples provide ESR-Al ages that are within error of each other for 3/4 of the samples considered in this study. These results show that ESR dating performed by the two independent laboratories yield generally reproducible chronologies.

628 5.1.2. <u>Ti-Li centre</u>

The Ti-Li ESR ages derived from the CENIEH samples provide an additional age 629 constraint for the deposits. In accordance with the basic principles of the MC approach, the Al 630 centre is considered to provide a maximum possible chronology for the deposits given the 631 differences observed in the D<sub>E</sub> estimates derived from each centre, while the Ti-Li chronology 632 most likely represents the closest estimate for the true age of sediment deposition. This, 633 however, does not mean that Al results measured in isolation (i.e., not as part of the MC 634 approach) should automatically be interpreted as systematically overestimating the true age of 635 636 the deposits. Recent studies have indeed demonstrated that this approach may sometimes provide ages in agreement with independent age control (e.g. Pereira et al, 2018; Duval et al 637 638 2015). Nevertheless, 3 out of 4 samples in the present study show overestimated Al ages, i.e.  $\geq$ 100 ka older than their Ti-Li equivalent (Fig. 5), while, interestingly, one sample from the 639 640 upper part of the sequence at BN site shows consistent Al and Ti-Li ESR ages (NOI1101, Fig. 5D). In this case, the combined Al-Ti-Li CENIEH age (Al:  $682 \pm 69$  ka; Ti:  $653 \pm 63$  ka) is also 641 in agreement with the ESR-Al MNHN samples from the same layer ( $680 \pm 77$  and  $699 \pm 76$ 642 ka). In contrast, sample NOI1104 at the bottom of the sequence yields an ESR-Al age of  $940 \pm$ 643 644 118 ka, which is about 200 ka older than the corresponding Ti-Li estimate (Fig. 4 & 5C). Here, in accordance with the principles of the MC approach, the difference given by the two centres 645

may be interpreted as evidence of the very limited transport experienced by the slope deposits, i.e. insufficient resetting of the Al signal due of its significantly slower bleaching kinetics (e.g. Duval et al., 2017a). The CENIEH Ti-Li age of  $739 \pm 72$  is also consistent at  $1\sigma$  with the MNHN ESR age of  $645 \pm 30$  ka from the same layer *a2* (Fig. 5C) and with the other 6 ESR ages from *b* above. These new results support the previous chronological assignment of ~700 ka for the Acheulean artefacts made by Moncel et al (2013).

At LTS, the Ti-Li ages provide an overall younger chronology (~650-730 ka) compared with the previous MNHN ESR-Al ages and the newly obtained CENIEH ESR-Al ages, which both indicated an Early Pleistocene chronology of around 800-1200 ka and 800-900 ka, respectively, for the sequence (Figs. 3, 5A & 5B). In accordance with the basic principles of the MC approach, the Ti-Li chronology is in the first instance considered to represent the closest estimate for the true age of sediment deposition. The accuracy of these results can, however, only be evaluated by cross-comparison with independent age control.

#### 659 5.2. Independent age control

The new single grain TT-OSL and palaeomagnetic data collected in the present study, 660 together with the TCN results published previously by Shen et al (2012), provide 661 stratigraphically well-constrained independent age control against which the ESR chronology 662 can be compared. Most notably, the ESR-Ti ages are consistent at  $1\sigma$  with the paired single-663 grain TT-OSL ages derived from closely associated samples at both sites:  $739 \pm 72$  ka (ESR-664 Ti) vs.  $644 \pm 47$  ka (TT-OSL) for BN layer *a*, and  $654 \pm 64$  ka (ESR-Ti) vs.  $727 \pm 51$  ka (TT-665 OSL) for LTS Unit 3. Despite their large associated uncertainties, the TCN ages of ~700-750 666 ka published by Shen et al (2012) are also consistent with the TT-OSL and ESR-Ti data. 667

In summary, the numerical ages obtained using three independent radiometric dating techniques collectively point to an early Middle Pleistocene chronology for the dated deposits at BN and LTS. These numerical chronologies are also supported by the palaeomagnetic data, which yielded a normal polarity for all of the samples collected at the two sites, and thus suggests that the deposits formed during the Brunhes chron (<772 ka; Okada et al., 2017).

### 5.3. Evaluating the impact of potential laboratory biases on the ESR age results

The absence of proper standardization regarding laboratory practices and dose rate evaluation in the field of ESR dating results in significant variability in the methodologies employed by different dating laboratories and research groups. Inter-laboratory comparisons performed on known-age samples and under well-controlled experimental conditions are 678 essential for examining any potential laboratory bias induced by the use of different methodologies. Unfortunately, such inter-laboratory comparisons are virtually non-existent 679 with regards to ESR dating of optically bleached quartz grains. The present work is perhaps 680 one of the closest attempts to undertake an inter-laboratory comparison study (see also Bahain 681 682 et al., accepted), although it should not strictly be considered as such, given the time elapsed between sampling by the two labs, and the random lateral distances between sampling positions 683 at each locality. Closer examination of the data produced by each laboratory shows that the 684 CENIEH samples display both lower D<sub>E</sub> and dose rate values compared to their MNHN 685 686 equivalents. However, any further data comparison is made complicated by the fact that these samples have been collected and processed in a totally independent way. In other words, it 687 should be kept in mind that the differences observed may be either due to local/lateral 688 variability within the sampled layer, or to the specific analytical procedure employed by each 689 laboratory. 690

691 In particular, although the CENIEH ESR samples and their MNHN equivalents can be 692 stratigraphically positioned within the same layers, they were not collected next to each other and there is thus an inherent uncertainty on the comparative dose rate and D<sub>E</sub> values. 693 694 Additionally, we have identified several differences in the analytical procedures employed and assumptions considered by the two laboratories. These differences are related to, among other 695 696 things, the internal dose rate (MNHN: null; CENIEH:  $50 \pm 30 \mu Gy/a$ ), the water content considered for the dose rate evaluation (MNHN: measured values between 10% and 15% (LTS) 697 698 or assumed to 10% (BN); CENIEH: assumed to  $20 \pm 5\%$ ), the *in situ* measurement of the 699 gamma dose rate (MNHN: systematic but posterior to the sampling; CENIEH: systematic and 700 at the same time as the sampling), and the choice of the fitting function used for the  $D_E$ 701 evaluation of the Al centre (MNHN: Single Saturating Exponential (SSE) for the LTS samples and EXP+LIN for the BN samples; CENIEH: EXP+LIN for all samples). Some of these 702 differences are naturally explained by the recent methodological developments that have taken 703 place since the publication of the MNHN ages (e.g. the systematic use of the EXP+LIN 704 function for the Al centre), while others are due to distinct conventions or practices applied by 705 706 each laboratory in the dose rate evaluation. It is beyond the scope of the present study to discuss the validity of each approach. However, sensitivity tests can nevertheless be performed to 707 evaluate their impact on the calculated ESR age results (Fig. 6). For example, the assumptions 708 709 around the internal dose rate carry very little weight in the total dose rate evaluation: when assumed to be null (like for the MNHN samples), the CENIEH ESR age results become slightly 710

older (by only 2%; Fig. 6). In comparison, when considering a water content of 10% for the
four CENIEH samples (i.e., similar to that used for most of the MNHN samples; Tables S1 &
S2), the ESR age estimates become younger by 12% on average; though they remain within
error of the original calculations (Fig. 6). These revised ages would still be in agreement with
the MNHN age results for 3/4 samples, and the overall age difference would even be reduced
for LUN1103, NOI1104 & NOI1101 (Fig. 5).

717

### Figure 6 approx. here

In summary, we have identified a series of differences between the methodologies employed by each laboratory. Although these differences have an apparent impact on the calculated ESR age estimates, they do not induce any statistically significant bias between the CENIEH and MNHN samples, which yield generally reproducible results.

722 5.4. Implications of the new dating results

The results of the present dating study provide significant advances for understanding the chronology of the two sites and the fluvial formations associated with the Cher River in general. At LTS, these results challenge current understanding of the site formation and stratigraphy, whereas at BN they strongly support previously published chronostratigraphic data.

727 5.4.1. Chronology of the fluvial formations associated with the Cher river

## 728 5.4.1.1. Unit 1 at LTS

729 The MNHN ESR-Al ages are consistent at  $1\sigma$  with both the new CENIEH ESR-Ti ages and the palaeomagnetic results, indicating a Brunhes age (< 772 ka) for the fluvial deposits of layer 730 b. In contrast, the ESR-Al age of  $893 \pm 87$  obtained for LUN1103 appears overestimated, most 731 likely as the result of incomplete resetting of the Al signal during transport. Consequently, a 732 mean age of  $792 \pm 57$  ka (n=3, 1 s.d.; Fig. 7) may be obtained for Unit 1 at LTS (Fig.7). This 733 result is consistent with the chronology of  $816 \pm 71$  ka (Despriée et al., 2011; see also Fig. 1), 734 previously established for fluvial terrace E (+29-34 m) (Despriée et al., 2011; see also Fig. 1). 735 However, the additional age constraint given by the palaeomagnetic data enables unequivocal 736 correlation of the fluvial deposits to the earliest part of the Middle Pleistocene, i.e. specifically 737 to a time interval of between 735 and 772 ka (considering the lower 1<sup>o</sup> uncertainty range of 738 the mean ESR age; Fig.7). Consequently, these results indicate that fluvial deposits at LTS 739 (Unit 1) may result from an aggradation phase around the interglacial/glacial transition 740 associated with MIS19-18. This is consistent with the model of river response typically 741

proposed for terrace system formation in north-west Europe (e.g. Antoine, 1994; Bridgland,
2000), which is mostly driven by cyclic climatic fluctuation.

744

### Figure 7 approx. here

### 745 *5.4.1.2.Unit 3 at LTS*

746 The present dating study sheds new light on the chronology of the fluvial deposits (layer b) of Unit 3 at LTS. Independent results derived from single-grain TT-OSL, TCN and ESR-Ti 747 methods are all consistent and provide a mean age of  $710 \pm 50$  ka (n=3; 1 s.d.). This combined 748 age estimate is within error of the chronology obtained for Unit 1 (735-772 ka) and consistent 749 750 with a correlation to MIS19-18 or MIS17-16 interglacial-glacial cyclicity. As detailed above (section 5.1), two main reasons may explain the significantly older ages previously obtained 751 752 for Unit 3 using the ESR-Al centre (~1.1-1.2 Ma; Despriée et al., 2011): the use of a different function to fit the experimental data points derived from the measurement of the Al centre, and 753 754 incomplete resetting of the ESR signal associated with the Al centre during sediment transport.

These new dating results raise many questions around the local chronostratigraphic 755 interpretation of the deposits. They indicate that the time interval between Unit 1 and 3 is 756 significantly shorter (20-60 ka) than initially assumed (>300 ka; Despriée et al., 2011). Units 1 757 to 3 have been initially correlated to three fluvial terraces E, F and G (e.g. Despriée et al., 2011). 758 However, given the uncertainty ranges of the new dating results (735-772 ka for Unit 1 and 759 760  $710 \pm 50$  ka for Unit 3), it can now be reasonably envisaged that the three units are almost coeval. Sedimentological evidence indicates that the three units are most likely not part of the 761 762 same depositional event, because they display very different sandy sedimentary facies (Unit 3: indurated coarse red sands with a high proportion of clays; Unit 2: horizontally laminated beige 763 764 sands typical of torrential flow; Unit 1: fine and laminated fluvial sands). The combination of these field observations with the newly obtained dating results suggest that the fluvial deposits 765 766 in each unit most likely correspond to different short aggradation events within the main aggradation phase associated with the formation of fluvial terrace E (e.g., Bridgland and 767 768 Westaway 2008).

Another hypothesis would be to consider that deposits from Unit 3 are indeed much older than those from Unit 1, but have been reworked and exposed to sunlight about 710 ka ago. However, the available chronological data do not support this possibility. The TT-OSL sample CRF11-2 does not show high D<sub>E</sub> overdispersion values that could indicate partial bleaching or different populations of grains with different bleaching history. Additionally, the independentage control provided by the TCN method also points towards a Middle Pleistocene chronology.

On the one hand, these unexpected young numerical dating results may contradict the initial 775 correlation of the sandy Units 1, 2 and 3 to the Sables-de-Rosières Formation, which delivered 776 the only fossil assemblage in the area at Lunery Rosières-Usine site (Despriée et al., 2017). Its 777 778 age, inferred from biochronology, is estimated to be around 1 Ma based on the similarities with other sites such as Le Vallonnet or Saint Prest (Guérin et al., 2003). Such an inference should 779 780 nevertheless be considered with caution, not only because of the inherent uncertainty associated with biochronology, but also due to the existing uncertainty in the taxonomic attribution of 781 some species, as mentioned by Guérin et al. (2003). However, on the other hand, stratigraphic 782 and sedimentological evidence at LTS suggests the presence of a major unconformity between 783 layers a and b (see Despriée et al., 2017a), which may indicate a significant chronological gap. 784 It is possible that the fluvial sediment originally deposited on top of a has been fully eroded 785 786 and replaced by much younger fluvial deposits from a subsequent aggradation phase. This 787 would explain the apparent discrepancy between the biochronology and the numerical age results obtained in the present study. However, it is difficult to test this hypothesis further, given 788 789 the absence of suitable material in layer a for numerical dating, and also because both Rosières-Usine and the LTS sites are no longer accessible. 790

### 791 *5.4.1.3 Layer b at BN*

At BN, all of the ESR results (CENIEH and MNHN, Al and Ti) obtained for the fluvial 792 deposits of layer b are consistent at  $1\sigma$ . They provide a mean age of  $665 \pm 29$  ka (n=8; 1 s.d.) 793 for fluvial terrace D (+14-21 m) of the Cher Valley (Fig.7), which is virtually the same as 794 published earlier ( $665 \pm 55$  ka, Despriée et al., 2011; see also Fig. 1). These combined dating 795 results indicate that the fluvial deposits of layer b accumulated around MIS 17-16, which is 796 consistent with the main aggradation phase defined by Bridgland (2000) at the interglacial-797 798 glacial transition. Alternatively, following the terrace formation system described by Antoine, (1994) for the nearby Somme valley, these fluvial deposits may be correlated to glacial 799 conditions prevailing during MIS16 (Despriée et al., 2017c). 800

801 5.4.2. <u>The age of the lithic assemblages at BN and LTS</u>

Because the lithic tools found at BN and LTS have not been directly dated by numerical means, the age of the hominin occupations must be inferred by integrating the existing numerical dating, sedimentological and stratigraphic evidence. To do so, a couple of key questions should be answered for each site: (i) to what extent are the artefacts found in *in-situ* position and have any been reworked from other deposits? (ii) to what extent can the numerical ages constrain the true age of the hominin occupation/presence ?

808 The study of the lithic assemblages at LTS and BN localities suggest that the artefacts underwent minimum transport and that both assemblages were found in primary position. 809 810 Prehistoric artefacts were knapped on siliceous blanks (millstone slabs and weathered Jurassic silicifications) gathered on the incision floor by hominins from the coarse slope deposits 811 coming from the plateau. The presence of cores, chopper-cores and flakes suggest that both 812 were workshop sites (e.g. Despriée et al., 2011). Additionally, the study of naturally 813 accumulated cobbles at BN has led to the identification of a series of features that are typical 814 of repeated high-energy fluviatile-type transport (Despriée et al., 2016). In contrast, the lithic 815 artefacts show knapped ridges that are not blunted and cutting edges that are not crushed, 816 suggesting that they experienced very little, if any, transport. Similarly, at LTS, the artefacts 817 818 show no evidence of surface abrasion as the result of a transport (Despriée et al., 2011). 819 Consequently, there is no evidence to suggest that archaeological levels should be interpreted as palimpsests. The data collected from fieldwork observations and laboratory analyses suggest 820 821 that the artefacts were protected from the subsequent glacial frost effects by the cover of new solifluction deposits, which were in-turn followed by the fluvial sandy deposits that currently 822 823 overlie the archaeological levels (Despriée et al., 2017a and b). The main question lies now in evaluating the time elapsed between the production of the lithic assemblages and the 824 subsequent accumulation of the slope and fluvial deposits. 825

At BN, the fluvial deposits associated with layer b provided a mean age of  $665 \pm 29$  ka, 826 which should thus be interpreted as a minimum age constraint for the Mode 2 lithic assemblage 827 found stratigraphically below, within layer *a*. Numerical dating of the slope deposits of layer 828 a2 post-dating the archaeological artefacts yields a mean age of  $690 \pm 52$  ka (n= 4: TT-OSL, 829 CENIEH ESR-Ti, MNHN ESR-Al and TCN; 1 s.d.). This result is stratigraphically consistent 830 with that of layer b. It provides an additional minimum age constraint for the lithic assemblage. 831 832 General interpretation of the site formation suggests that this accumulation was deposited after 833 the end of river incision, i.e. at the beginning of a glacial period based on the model proposed by Antoine (1994) for the nearby Somme valley, and locally underwent cryoturbation together 834 835 with the altered limestone level during the glacial maximum (Despriée et al., 2007b). Consequently, given the existing age uncertainty, the beginning of MIS16 around 676 ka 836 837 (Lisiecki and Raymo, 2005) may provide a maximum age constraint for the diamicton at the

bottom of the sequence, while the 1- $\sigma$  upper range of the mean age (690-52 = 638 ka) may be used as a minimum age constraint. Therefore, an age range of 676-638 ka may be proposed for the hominin occupation at BN (Fig. 7). This result is consistent with the initial chronology proposed by Moncel et al (2013) and confirms that BN is among the oldest Acheulean occurrences in Western Europe north of the 45°N latitude (see also Antoine et al., 2019).

At LTS, the mean age of  $710 \pm 50$  ka obtained for the sandy layer b provides a minimum 843 age constraint for the archaeological level located below. Despriée et al., (2017a) position the 844 hominin presence at the end of the incision phase, which might have occurred during the 845 earliest part of MIS 18 following the model by Antoine (1994). However, the detailed 846 stratigraphic profile of the excavation suggests a major disconformity between the pebble 847 layers hosting the lithic artefacts within a (labelled c0, c1 and c2 in Despriée et al., 2017a) and 848 the fluvial deposits associated with layer b. Consequently, it may reasonably be envisaged that 849 deposits from layers a and b were not deposited during the same aggradation phase. If so, then 850 851 the chronological gap between these layers might be significant, which would imply that the 852 estimated age of the lithic assemblage at LTS is much older than  $710 \pm 50$  ka. Indeed, the major technological and typological differences between the lithic assemblages at LTS and BN might 853 854 also be considered as indirect evidence to support this hypothesis. At LTS, the lithic assemblage is made of choppers, chopper-cores on cobbles with small flakes (Mode 1) and without bifacial 855 856 pieces or large cutting tools. In contrast, the assemblage from BN contains only cores on millstone slabs with flakes, and numerous Acheulean handaxes and large cutting tools (Mode 857 2). These significant differences between both assemblages is more likely to be explained by a 858 major chronological gap between the two occupations than by the opportunistic behaviour of 859 860 hominins in the earliest part of the Middle Pleistocene. Technological and typological characteristics of the lithic artefacts found at LTS fall within the variability of the Mode 1 861 assemblages described at the Early Pleistocene sites of Europe (see section 6.3 in Despriée et 862 al., 2017a). Consequently, a comparable minimum age of 0.8 Ma may reasonably be inferred 863 for the lithic assemblage of Unit 3 at LTS, which is consistent with the minimum age of  $710 \pm$ 864 50 ka suggested in the present work. Cryoturbation features affecting the accumulation of 865 blocks, cobbles and pebbles at the bottom of Unit 3 document the occurrence of a cold climate 866 that could be tentatively linked to a glacial stage of the late Early Pleistocene (e.g., MIS20, 867 MIS22?). However, in the absence of direct age control for the deposits of layer a, there is, for 868 the moment, no clear evidence supporting a correlation of hominin presence at LTS to a given 869

MIS. Any further attempts at refining the chronological constraint of Unit 3 would become toospeculative.

872

## 873 Conclusion

The dating results of the present study have not only helped to further constrain the 874 chronology of the fluvial deposits from the Cher river, one of the Loire tributaries, but they 875 have also shed new light on the age of two key Lower Palaeolithic sites located in central 876 877 France: Lunery-La Terre-des-Sablons and Brinay-la Noira. Hominin presence at BN may tentatively be correlated to the early MIS16 (676-638 ka), which is in agreement with previous 878 interpretations. In contrast, only a minimum age of ~710 ka may be securely proposed for 879 hominin presence at LTS, although an Early Pleistocene chronology may not be discounted 880 based on stratigraphical and technological evidence. Unfortunately, any future attempt to obtain 881 additional chronological constraints is almost impossible as the site is no longer accessible. 882

The present study is another example showing the importance of using the Multiple Centre 883 884 approach in ESR dating of quartz (e.g. Duval et al., 2017a; Kreutzer et al., 2018; Mendez-Quintas et al., 2018), which remains the best way to evaluate potential incomplete resetting of 885 886 the ESR-Al signal. Finally, given the inherent non-negligible uncertainties associated with the use of dating methods such as ESR, luminescence and TCN, the multi-technique dating 887 approach used here is undoubtedly the most appropriate way to build more robust and accurate 888 chronologies for Early to Middle Pleistocene archaeological sites found in fluvial sedimentary 889 890 environments. The conditions needed for applying this type of multi-technique dating approach are frequently met at Lower Palaeolithic sites located in the Mediterranean region, providing 891 an invaluable means of maximising dating reliability in contexts where highly standardized 892 and accurate methods (e.g., radiocarbon, U-series or argon-argon dating) cannot be routinely 893 employed. 894

From a methodological point of view, the dating results presented here are especially encouraging for the reliability of the ESR method applied to optically bleached quartz grains. This is to our knowledge one of the first studies demonstrating that ESR quartz samples independently collected, processed and dated by two different laboratories may produce reproducible ESR age results. This kind of comparative work is essential to strongly position the ESR method as a reliable alternative numerical dating method in Quaternary studies.

### 902 Acknowledgement

903 The study was funded by project CGL2010-16821 from the Spanish Ministry of Science and Innovation and Australian Research Council (ARC) Future Fellowship Grant 904 905 FT150100215. Walter Minnella's research stays at CENIEH were covered by the Lifelong Learning Programme Erasmus Placement program granted to University of Catania, and the 906 EARTHTIME-EU - Exchange Grant #4569 from the European Science Foundation. Additional 907 financial support for the TT-OSL dating research was provided by Australian Research Council 908 (ARC) Future Fellowship project FT130100195 and ARC Discovery Early Career Researcher 909 Award DE160100743. Carlos Pérez Garrido is thanked for his assistance with preparing and 910 measuring the luminescence dating samples at the CENIEH luminescence dating laboratory, 911 Burgos, Spain. Finally, the authors would like to thank the three anonymous reviewers for their 912 constructive comments, which have contributed to significantly improve the quality of the 913 914 manuscript.

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1178	Runcorn, S.K. and Creer, K.M. (Eds.), Methods in Paleomagnetism. New York: Elsevier.
1179	pp. 254-286.

### 1183 **Table caption**

1184Table 1: ESR data derived from the measurement of the Al centre. Measurement repeatability1185is expressed as the variation (one relative standard deviation) of the mean ESR intensity1186obtained from all the aliquots of a given sample after each day of measurement. Similarly, the1187repeatability of the  $D_E$  values corresponds to the variation (one relative standard deviation) of

1188 the  $D_E$  values calculated for each day of measurement. Bleaching coefficient (%) is defined as

- the relative difference between the ESR intensities of the natural and bleached aliquots.
- 1190 Table 2: ESR data derived from the measurement of the Ti centre. Measurement repeatability1191 is expressed as the variation (one relative standard deviation) of the mean ESR intensity

1192 obtained from all the aliquots of a given sample after each day of measurement. Similarly, the

1193 repeatability of the D<sub>E</sub> values corresponds to the variation (one relative standard deviation) of

- the  $D_E$  values calculated for each day of measurement.
- 1195 Table 3: Detail of ESR age estimates and dose rate components. Errors are 1 sigma.
- 1196 Table 4: TT-OSL dose rate data, single-grain equivalent doses (D<sub>E</sub>) and final ages for samples
- 1197 CRF11-2 and CRF11-4. The final TT-OSL ages have been derived by dividing the weighted
- 1198 mean  $D_E$  by the total dose rate.
- Table 5: Summary of paleomagnetic data. Dec / Inc: Declination and Inclination of the
  Characteristic Remanent (ChRM) Magnetization direction; MAD: Maximum angular
  deviation; Plat / Plong: Latitude and longitude of the Virtual Geomagnetic Pole (VGP) position.
  Samples LUN1 and NOR1-6 did not vield conclusive (n.c.) results.

1203

### 1205 **Figure caption**

Figure 1. A: Geological map of the Centre Region (France) including the middle Loire Basin and junction of the River Loire River and its main tributaries (Loir, Cher and Creuse) (modified from Despriée et al., 2011). The circles indicate the Palaeolithic sites identified in the three valleys. Key: 1 = Lunery-la Terre-des Sablons ; 2 = Brinay-la Noira ; 3=Gievres-la Genetière ; 4 = Gièvres-la Morandière; 5 = Gièvres-la Plaine-de-la-Morandière. B: Synthetic chronostratigraphy of the Cher valley terasse system (Despriée et al., 2011).

Figure 2: Geological context of Lunery-la Terre-des Sablons (LTS) site in the so-called "Les Sablons" block, which is delimited to the west and east by the Rosières fault and Cher river channel, respectively. Local subsidence phenomena have affected the block, which has been successively stripped in various subsets (as indicated by vertical arrows labelled 1, 2 and 3). This interpretation is based on field observations, sedimentological and geochronological evidence collected before the present study, as summarized in section 1.2 and in Despriée et al., (2007a). The position of the archaeological excavation is indicated.

Figure 3: Stratigraphic column of Lunery-la Terre-des Sablons site (modified from Despriée et 1219 1220 al., 2007a). The position of all the samples (and their corresponding age results) collected for dating purpose is indicated (Red = ESR sample MNHN; Green = ESR sample CENIEH; Purple 1221 = TT-OSL sample; Blue = palaeomagnetic sample; Yellow = TCN sample). TCN and MNHN 1222 ESR age results have been previously published in Shen et al. (2012) and Despriée et al., 1223 (2007a) (and references therein), respectively. Dose rate and ESR age calculation details may 1224 be found in Supplementary Information, Tables S1. Colours are available in the pdf version of 1225 1226 the paper.

1227 Figure 4: Stratigraphic column of Brinay-La Noira site (modified from Moncel et al., 2013). The position of all the samples (and their corresponding age results) collected for dating 1228 purpose is indicated (Red = ESR sample MNHN; Green = ESR sample CENIEH; Purple = TT-1229 OSL sample; Blue = palaeomagnetic sample; Yellow = TCN sample). Three MNHN ESR 1230 1231 samples have not been published elsewhere earlier. The other TCN and MNHN ESR age results have been previously published in Shen et al. (2012) and Despriée et al., (2011), respectively. 1232 1233 Dose rate and ESR age calculation details may be found in Supplementary Information, Tables S2. Colours are available in the pdf version of the paper. 1234

Figure 5: Comparison of the ESR ages obtained by MNHN and CENIEH at Lunery-la Terre-des Sablons and Brinay-La Noira sites. Results are grouped per layer. Corresponding numerical

- values can be found in Supplementary Information, Tables S1 and S2. Colours are available inthe pdf version of the paper.
- 1239 Figure 6: Sensitivity tests performed on the four CENIEH samples to evaluate the impact of
- internal dose rate and water content assumptions on the ESR age results derived from the Al
- 1241 centre. Key: black circles = initial age calculation (Fig. 5), with internal dose rate =  $50 \mu Gy/a$
- and water content = 20%; red triangles = ESR age results with internal dose rate =  $50 \mu Gy/a$
- 1243 and water content = 10%; Green squares = ESR age results internal dose rate =  $0 \mu Gy/a$  and
- 1244 water content = 20%. Colours are available in the pdf version of the paper.
- 1245 Figure 7: Graphical summary of the chronologies obtained in the present work for the fluvial
- 1246 and slope deposits, as well as for the human presence at LTS and BN localities. MIS data are
- 1247 from Lisiecki & Raymo (2005)

# **Table 1.**

Sample	Number of measurement	Bleaching Coefficient (%)	Repeatability of the ESR intensities (%)	$\begin{array}{c} \text{Repeatability} \\ \text{of the } D_{\text{E}} \\ \text{estimates} \\ (\%) \end{array}$	Adjusted r <sup>2</sup>	D <sub>E</sub> value (Gy)
LUN1101	3	$58.0 \pm 1.8$	1.8	7.0	0. 993	$2560 \pm 204$
LUN1103	5	$55.7 \pm 1.6$	7.4	12.7	0. 993	$2391 \pm 129$
NOI1101	3	$50.7 \pm 0.7$	1.4	7.3	0.994	$1788 \pm 113$
NOI1104	3	$57.4 \pm 1.9$	3.4	8.6	0.989	$2639\pm256$

# **Table 2.**

Option	Α		D				E	
Sample ID	Adj. r <sup>2</sup>	$D_E(Gy)$	Measurement repeatability (%)	D <sub>E</sub> repeatability (%)	Adj. r <sup>2</sup>	$D_E(Gy)$	Adj. r <sup>2</sup>	$D_E(Gy)$
LUN1101	0.991	2688 ± 117	0.5	8.5	0.987	1934 ± 101	0.993	2802 ± 113
LUN1103	0.987	2143 ± 98	1.1	15.4	0.987	1951 ± 99	0.988	2660 ± 114
NOI1101	0.991	$\begin{array}{c} 1700 \pm \\ 63 \end{array}$	1.3	8.6	0.995	1712 ± 93	0.993	2270 ± 50
NOI1104	0.992	2357± 113	0.8	16.0	0.986	$2074 \pm 109$	0.994	3078 ± 214

## **Table 3.**

Sample	LUN1101	LUN1103	NOI1101	NOI1104
Unit / Layer	3	1	b	а
Depth (m)	12±3	3.5±0.5	1.5±0.5	7±1
Measured water content (% wet weight)	8.4	7.1	9.8	12.1
Assumed water content (% wet weight)	20±5	20±5	20±5	20±5
Internal dose rate (µGy/a)	50 <u>+</u> 30	50 <u>+</u> 30	50 <u>+</u> 30	50 <u>+</u> 30
Alpha dose rate (µGy /a)	23 <u>+</u> 20	28 <u>+</u> 24	44 <u>+</u> 39	27 <u>+</u> 23
Beta dose rate (μGy/a)	2019 <u>+</u> 177	1639 <u>+</u> 143	1414 <u>+</u> 120	1879 <u>+</u> 162
Gamma dose rate (μGy/a)	821 <u>+</u> 75	839 <u>+</u> 77	948 <u>+</u> 87	781 <u>+</u> 71
Cosmic dose rate (µGy/a)	44 <u>+</u> 15	121 <u>+</u> 11	166 <u>+</u> 16	72 <u>+</u> 10
Total dose rate (μGy/a)	2957 <u>+</u> 238	2677 <u>+</u> 209	2622 <u>+</u> 200	2807 <u>+</u> 221
D <sub>E</sub> (Gy) Al centre	2560 <u>+</u> 212	2391 <u>+</u> 141	1788 <u>+</u> 121	2639 <u>+</u> 263
D₌ (Gy) Ti-Li centre	1934 <u>+</u> 110	1951 <u>+</u> 108	1712 <u>+</u> 101	2074 <u>+</u> 119
Age (ka ) Al centre	866 <u>+</u> 100	893 <u>+</u> 87	682 <u>+</u> 69	940 <u>+</u> 119
Age (ka ) Ti-Li centre	654 <u>+</u> 64	729 <u>+</u> 70	653 <u>+</u> 63	739 <u>+</u> 72

1256 **Table 4** 

-				Grain	Long-term	Environmental dose rate (Gy/ka)				Equivalent dose (D <sub>E</sub> ) data				
	Sample	Site / Unit (µm)	size (µm)	size water (µm) content <sup>a</sup>	Beta dose rate <sup>b,c</sup>	Gamma dose rate <sup>c,d</sup>	Cosmic dose rate <sup>e</sup>	Total dose rate <sup>f,g</sup>	No. of grains <sup>h</sup>	Overdis- persion (%) <sup>i</sup>	Age Model <sup>j</sup>	D <sub>E</sub> (Gy) <sup>f</sup>	age (ka) <sup>f,k</sup>	
_	CRF11-4	BN	а	125 – 180	22 ± 4	1.88 ± 0.10	0.85 ± 0.03	0.08 ± 0.01	2.85 ± 0.18	181 / 1900	34 ± 5	CAM	1833 ± 58	644 ± 47
	CRF11-2	LTS	3	125 – 180	21 ± 4	2.08 ± 0.11	$0.85 \pm 0.03$	0.05 ± 0.01	3.02 ± 0.18	195 / 2000	29 ± 5	CAM	2194 ± 63	727 ± 51

#### 1257

1258 <sup>a</sup> Long-term water content, calculated as 60% of the present-day saturated water content and expressed as % of dry mass of mineral fraction, with an assigned relative

1259 uncertainty of  $\pm 20\%$ .

<sup>b</sup> Calculated on dried and powdered sediment samples using a Risø GM-25-5 low-level beta counter.

1261 <sup>c</sup> Specific activities and radionuclide concentrations have been converted to dose rates using the conversion factors given in Guérin *et al.* (2011), making allowance for

1262 beta-dose attenuation (Mejdahl, 1979; Brennan, 2003).

<sup>d</sup>Calculated from *in situ* measurements made at each sample position with a NaI:Tl detector, using the 'energy windows' approach (e.g., Arnold *et al.*, 2012b).

<sup>e</sup> Cosmic-ray dose rates were calculated using the approach of Prescott and Hutton (1994), and assigned a relative uncertainty of ±10%.

1265 f Mean  $\pm$  total uncertainty (68% confidence interval), calculated as the quadratic sum of the random and systematic uncertainties.

- 1266 <sup>g</sup> Includes an internal dose rate of 0.03 Gy/ka with an assigned relative uncertainty of  $\pm 30\%$ .
- <sup>1267</sup> <sup>h</sup> Number of D<sub>E</sub> measurements that passed the SAR rejection criteria and were used for D<sub>E</sub> determination / total number of grains analysed.

<sup>1268</sup> <sup>i</sup> The relative spread in the D<sub>E</sub> dataset beyond that associated with the measurement uncertainties for individual D<sub>E</sub> values, calculated using the central age model (CAM)

1269 of Galbraith *et al.* (1999).

<sup>j</sup> The CAM was used to calculate the final D<sub>E</sub> as these samples had overdispersion values consistent with those observed in 'ideal' well-bleached and unmixed sample

- 1271 from similar settings (Arnold and Roberts, 2009; Arnold et al., in press).
- 1272 <sup>k</sup> Total uncertainty includes a systematic component of  $\pm 2\%$  associated with laboratory beta-source calibration.

Specimen	Stratigraphic position	Dec	Inc	MAD	Plat	Plong
Lunery						
LUN2-1A	Unit 1	.8	55.7	5.4	79	179
LUN2-2A	Unit 1	2.1	64.4	2.5	88	120
LUN2-3A	Unit 1	6.7	69.2	10.6	83	36
LUN2-1B	Unit 1	2.6	59.4	7.7	83	166
LUN2-2B	Unit 1	10.4	62.8	4.8	82	109
LUN2-2D	Unit 1	2.5	64.5	12.4	88	112
LUN2-3C	Unit 1	359.0	69.1	7.0	84	356
LUN1	Unit 3	n.c.	n.c.	n.c.	n.c.	n.c.
La Noira						
NOR1-1.1	Laver a (North section)	349.8	47.3	15.8	73	312
NOR1-1.4	Layer a (North section)	7.2	51.1	14.0	70	209
NOR1-2.1	Layer a (North section)	2.8	69.2	20.3	84	18
NOR1-2.3	Layer a (North section)	13.2	59.6	11.9	82	151
NOR1-3.3	Layer a (South section)	17.9	56.7	9.0	82	214
NOR1-3.2	Layer a (South section)	5.2	59.5	7.4	84	237
NOR1-4.1B	Layer a (South section)	354.4	59.2	7.8	81	359
NOR1-4.2C	Layer a (South section)	353.2	62.1	7.5	78	162
NOR1-4.3B	Layer a (South section)	359.1	71.6	15.6	74	159
NOR1-4.1A	Layer a (South section)	.4	59.1	9.6	78	122
NOR1-4.2B	Layer a (South section)	345.2	63.9	11.0	74	122
NOR1-4.3C	Layer a (South section)	349.8	68.2	8.0	83	179
NOR1-5.2	Layer a (South section)	4.9	55.1	12.6	80	269
NOR1-5.4	Layer a (South section)	349.8	47.3	15.8	82	0
NOR1-6	Layer b (North section)	n.c.	n.c.	n.c.	n.c.	n.c.

## **Table 5.**















