PALEO
Revue d'archéologie préhistorique

23 | 2012
Varia

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OpenEdition Journals

Electronic version
URL: http://journals.openedition.org/paleo/2466
DOI: 10.4000/paleo.2466
ISSN: 2101-0420

Publisher
SAMRA

Printed version
Date of publication: 15 December 2012
Number of pages: 155-170
ISSN: 1145-3370

Electronic reference
Marion Hernandez, Norbert Mercier, Pascal Bertran, David Colonge and Laure Amélie Lelouvier, « First dating results for the Middle Pleistocene industries (Acheulean – Early Middle Palaeolithic) in the Pyrenees – Garonne region: a multi methods geochronological approach (TL, OSL and TT-OSL) of the Duclos and Romentères sites », *PALEO* [Online], 23 | 2012, Online since 03 June 2013, connection on 27 July 2020. URL : http://journals.openedition.org/paleo/2466 ; DOI : https://doi.org/10.4000/paleo.2466

First dating results for the Middle Pleistocene industries (Acheulean – Early Middle Palaeolithic) in the Pyrenees – Garonne region: a multi methods geochronological approach (TL, OSL and TT-OSL) of the Duclos and Romentères sites

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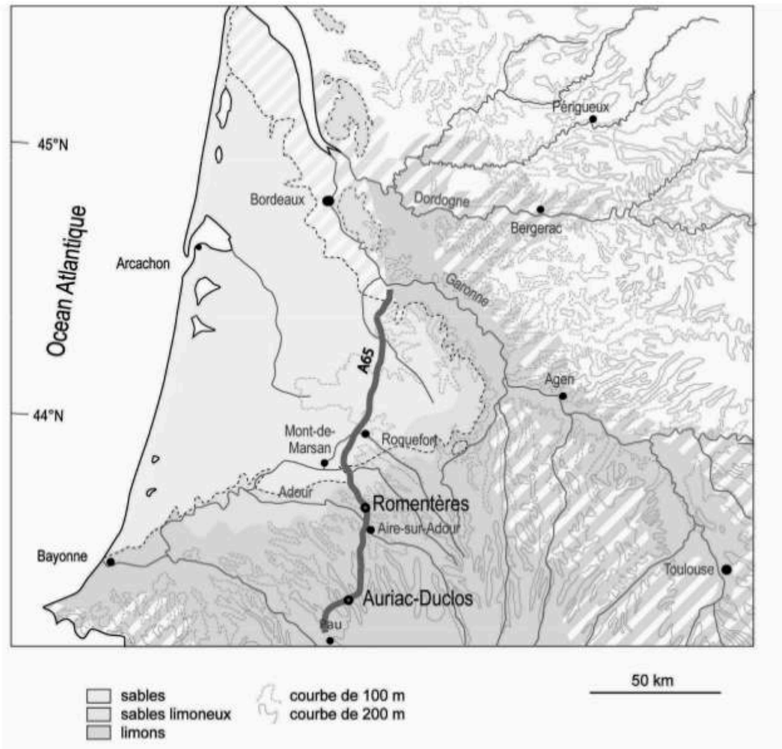
1- Introduction

1 In recent years, research on lithic assemblages of the Aquitaine basin prior to MIS 5 has broadened considerably in part because of a new manner to look at the anciently excavated series (Mourre and Colonge 2007), but also thanks to recent discoveries mostly due to archaeological rescue works (Bourguignon *et al.* 2008; Brenet 2011; Colonge 2005; Jarry 2010). The diversity at a regional scale of the Lower and Middle Palaeolithic industries has long been emphasized (Bordes 1971; Villa 1981; Jaubert and Servelle 1996; Colonge and Mourre 2009). In this context the techno-cultural originality of the Acheulean of the Pyrenees-Garonne region, characterised by chaînes opératoires of large blank production (over 15 cm) and by the presence of cleavers *sensu stricto*, has been highlighted (Mourre and Colonge *op. cit.*; Colonge *et al. in press*). Recent works on the A65 motorway connecting Langon to Pau has allowed renewal of the available data on this techno-complex, also exposing an abundant series attributed to the Early Middle Palaeolithic in Romentères (fig. 1). These discoveries have given the opportunity through the cross analysis of the different assemblages, to discuss the

relations between the Acheulean and the Early Middle Palaeolithic in the western part of the Pyrenean Piedmont. Although the techno-typological approach of these assemblages shed light on the technical and economical, and even cultural behaviours, their comparison has often been limited by an overall lack of chronological data (Turq *et al.* 2010). Indeed, if some French regions such as the North presently have a relatively well defined chronostratigraphic framework (Antoine *et al.* 2003a, 2007, 2011), the Southwest has long been considered unsuitable for this type of synthesis because of the local characteristics of the sedimentation and the lack of reference stratigraphic sequences. However, recent rescue excavations carried out on the A65 highway have greatly contributed to overcoming this gap. These new data have highlighted the presence along the future highway of a thick silty cover that comprises palaeosoils: their deposition was conditioned by global climatic variations that affected the northern hemisphere, just as with the sequences of Northern Europe (Bertran *et al.* 2009, 2011). In order to build thanks to this favourable context a chronostratigraphic framework in which the industries discovered in these deposits will fit, a dating campaign by luminescence methods was carried out on the sites of Romentères (Le Vignau, Landes) and Duclos (Auriac, Pyrénées-Orientales). In this study, it was decided to date both the heated lithics by thermoluminescence (TL) as well as the sediments by Optically Stimulated Luminescence (OSL) in order to meet several goals: to place the lithic industries chronologically; to date the palaeosoils and use them as chronostratigraphic reference points ; and to reflect on the taphonomic history of the sites by comparing the TL ages of the heated lithics to the OSL ages of the surrounding sediments.

2 Given the antiquity of the sampled deposits (Middle and Upper Pleistocene), the dating of the sedimentary quartz necessitated the development of a new methodology based on the use of an OSL signal obtained through thermal transfer (TT-OSL). Preliminary results have already been published concerning the methodology developed for this signal (Hernandez *et al.* 2012), and the Aeolian deposits of Southwest France (Bertran *et al.* 2011). The entirety of the chronological data obtained for the sites of Duclos and Romentères has also been used in the context of an archaeological synthesis of the Middle Pleistocene occupations of the Western Pyrenean Piedmont (Colonge *et al.* SPF, in press). However, we thought it was important to present here in more detail the acquisition of the results, discuss their validity and also detail the chronostratigraphic results and their implications.

Figure 1 - Map of aeolian deposits of the Aquitaine region and geographical location of Duclos and Romentères sites (after Bertran et al. 2011).



2 - Description of sequences

2.1 – Stratigraphy

3 The sequences observed at the Duclos and Romentères sites are rather similar and include the following pedostratigraphic units, from top to bottom:

4 An upper ploughing horizon (LA), brown, 35 cm thick on average;

5 An eluviated silty (E) horizon, light brown, 5 to 30 cm thick, with a glossic lower boundary. In a plan view, the tongues make a semi-metric polygonal network that originates from cryogenic processes (Van Vliet and Langohr 1981). The presence of residual clayey aggregates, often strongly impregnated by Fe and Mn oxides, at the base of horizon E shows that it has deepened progressively during the Holocene at the expense of the Bt horizon, following leaching and alteration of the clay particles along the cracks;

6 3) An illuvial horizon (BTgx), yellow brown (10YR), 0.5 to 1 m thick. A coarse platy structure inherited from segregation ice lenses (Van Vliet-Lanoë 1985) is usually visible from 1 m deep. This horizon developed on aeolian loess deposits (granulometric mode around 30 µm) that make a nearly continuous cover on the plateaus in the region (“upper silts”). The lower part of this layer comprises scattered small gravels that indicate a colluvial origin of the sediment (a mixture of products coming from the dismantling of the alluvial substrate and from earlier loess cover). These colluvial deposits are locally cryoturbated ;

7 A buried illuvial horizon (IIBTg), bright brown (7.5YR) 0.5 to 1 m thick. The upper limit shows tongues and is locally rich in Fe-Mn concretions. Vertical and horizontal bleached lenses, coming from former segregation ice lenses and blades, make a characteristic reticulated pattern. This horizon developed on loess and silty colluvial deposits with some gravels (“middle silts”);

8 A second buried illuvial horizon (IIIBTg) only seen at Duclos, red brown (5YR) and grey, about 0.5 m thick, on silty colluvial deposits with scattered gravel (“lower silts”). Like in the previous horizon, bleached lenses form a reticulated pattern. The base of this layer is locally underlined by a gravel bed;

9 A third illuvial horizon, seen at both sites, (IV BTg) red (2.5YR) and grey, banded, developed on coarse, strongly weathered alluvial gravel at Duclos and on clayey alluvial deposits at Romentères.

10 In total the sequence shows two (Romentères) or three (Duclos) silty units (loess and silty colluvial deposits) separated by palaeosols (luvisols) and gravel-rich layers which locally form pavements. These palaeosols testify to soil-forming periods similar to the present and are thus considered as interglacial ranking. They each show distinct characteristics, in their colour as well as by the clayey illuviation. SThe redder their colour is, the older they are (IIBTg: 7.5 YR, IIIBTg: 5YR, IVBTg: 2.5YR) probably because of progressive transformation of the oxides through time resulting in the formation of less hydrated minerals (Duchaufour 1983; Cremaschi 1987). The chronological hypothesis proposed by the lithostratigraphic study suggests an Eemian age (last interglacial, MIS 5) for horizon IIBTg, while horizon IIIBTg would correspond to the previous interglacial (MIS 7). The underlying red palaeosol, which developed on strongly weathered alluvial deposits is thought to reflect the pedosedimentary budget of several interglacials of the Lower and Middle Pleistocene.

Figure 2 - General views of Duclos and Romentères stratigraphies. The scale represents 1 m. Duclos: the red-brown paleo argilic horizon (IIIBtg) can be seen at the base of the trench while the layer of alluvial gravels is not visible in this picture. Romentères: the ploughed soil is not visible because it was previously stripped.

2.2 - Archaeological levels

11 The site of Duclos (Colonge dir., pending) has yielded a characteristic lithic series of the Pyrenees-Garonne Acheulean, very close to the Acheulean of Iberian type (Colonge and Texier 2005; Mourre and Colonge 2007.) In particular this industry features a specific production of large blanks, a distinctive bifacial fashioning (mostly nucleiform) and the fashioning of cleavers and polyhedrons. The remains are for a third of them *in situ* (residual concentrations) in the “middle silts” (IIBTg). However, most of the lithics, over two thirds of them, form a diffuse layer in the “upper silts” (BTgx).

12 At the Romentères site, two series, clearly distinct from a techno-cultural point of view and unequal in terms of quantity of material and conservation state, were reported (Lelouvier *et al.* in press). The most important collection, with over 4,500 objects, is densely spread in the upper part of the “middle silts” (IIBTg) and comes partly (about 15 %) from the “upper silts”. The collection is fresh looking, and contains numerous small elements. This series corresponds to the Early Middle Palaeolithic and contains rare bifaces similar to its counterparts in the Aquitaine basin (Jarry *et al.* 2007; Bourguignon *et al.* 2008; Colonge *et al.* 2010). This assemblage is characterised by a marked economy of raw materials, the systematization of the Levallois debitage, an emphasis on scrapers, classic bifacial fashioning and the strict absence of cleavers. The second series only contains 200 objects, clearly sorted out (over 5 cm in size) and strongly altered (deep alteration and strong wear of the edges). The “nucleiform” bifaces, cleavers and the polyhedrons indicate a Pyrenees – Garonne Acheulean facies

similar to the one at Duclos. These remains are spread within layers containing the Early Middle Palaeolithic in the "middle silts."

3 - Geochronologic data

3.1 - Principle of luminescence dating methods

13 Luminescence dating methods are based on the capacity of minerals such as quartz to accumulate energy from radioactive origin in their crystalline defects in the form of trapped electrons. The energy is released via the production of light when the minerals are exposed to optical stimulation (which is called Optically Stimulated Luminescence, OSL) or a thermal one (thermoluminescence, TL).

14 Minerals are constantly exposed to radiations as they contain and are surrounded by natural radioelements : the Potassium (K-40) and the families of Uranium 238 (U-238) and Thorium 232 (Th-232). In disintegrating, these radioelements and their descendants produce gamma rays as well as alpha and beta particles. Cosmic rays are also a source of radiation exposure for the material. These forms of radiation ionize the atoms and free electrons, some of which are captured by the traps associated to the defects in the crystalline lattice. Thus, through time, the number of trapped electrons increases continuously in the dating material.

15 Since these radiations are, on the whole, constant through time, the relation between the elapsed time and the quantity of trapped electrons is well established. Thus, to date, it is necessary to determine the total radiation dose received by the sample (usually called palaeodose). It is also necessary to determine the associated dose rate, i.e. the dose received per time unit. The ratio of these two parameters then gives the age of the sample, i.e. the time elapsed since the last resetting of the sample (Aitken 1985). From this point of view, the TL, OSL or TT-OSL dating methods are strictly identical in their principle and only differ in the type of signals used to determine the palaeodose.

16 It is important to note that the age resulting from the application of these methods corresponds to the dated event only if the previously accumulated dose had been entirely erased in the past (because of an archaeological heating in the case of burnt stones, or during the last exposure to light in the case of sedimentary quartz) ; otherwise the age of the sample would be overestimated. This condition can be checked in the case of heated rocks, but no preliminary test can ensure this condition was satisfied in the case of quartz grains "bleached" by light. Specific attention has thus been given to this issue in the present study.

3.2 - Dating of sedimentary quartz (OSL, TT-OSL)

3.2.1 - Sampling and preparation of samples

17 From a practical point of view, each sediment sample was collected by inserting an opaque tube (made of PVC or Duraluminium) into the stratigraphic section, so that the quartz grains were not exposed at any time to day light. A further fraction of sediment was also systematically collected (with less precaution with regard to light exposure) in order to determine later in the laboratory its radioisotopic contents (K, U, Th).

18 Prior to the preparation of the sample fraction used for luminescence measurements, the external 2 cm of sediment in the sampled tube, likely containing grains exposed to light during sampling, were removed. The fraction of grains comprised between 20 and 41 µm, most abundant in the samples of Duclos and Romentères, was extracted by a water sieving. A chemical treatment was then performed to extract and purify the quartz grains. To this end, the following process was applied: a first cleaning with hydrochloric acid (HCl) allowed dissolution of the carbonates; then a five-day treatment with oxygenated water (H_2O_2) was done to eliminate the organic matter; finally, the feldspar grains were eliminated through the use of a hexafluorosilicic acid (H_2SiF_6) solution applied for 7 days. Eliminating feldspars is essential because their luminescence signal can easily contaminate the signals emitted by the quartz. The efficiency of this last chemical treatment has been assessed by making sure of the absence of any luminescence feldspar signal, through the stimulation with infra-red light of a fraction of each studied sample.

3.2.2 - Determination of the dose rate

19 The radioisotopic (K, U, Th) contents of the sediments have been determined in the laboratory by low background gamma-ray spectrometry. No disequilibrium in the uranium chain was detected, what suggests that the doses deduced from this series did not change significantly through time. Furthermore, the obtained results indicate similar radioisotopic contents in each site, for the Duclos and Romentères samples, and thus a good homogeneity of the radioactive environments (tabl. 1). The beta, alpha and gamma dose rates have been calculated from these data in considering the conversion factors published by Adamiec and Aitken (1998). It is usually preferable to measure on site the gamma dose rate, what was not possible in the two cases presented here. However, the gamma doses deduced from the laboratory measurements performed on the sampled sediments have been considered as valuable for the calculation of the ages because of the radioactive homogeneity of the sediment samples

20 For each sample, the cosmic dose rate has been assessed taking into account the samples depth at present and through time, and considering the values tabulated by Prescott and Hutton (1994). Considering the difficulty of assessing this parameter precisely, a 10% uncertainty has been added. However, thanks to the relatively small contribution of this component to the total dose rate (around 8 %), its impact on the age remains low (tabl. 1).

21 Finally, water contents of 12 % for the Romentères samples and 14 % for Duclos have been taken into account. These values correspond to the average humidity contents measured in the laboratory for the collected samples. Because of the impossibility of measuring the variation of sediments' humidity through time, an uncertainty of 10 % has been assigned to this parameter. Given the influence of humidity on dose rates, this data is one of the main sources of uncertainty on the ages.

3.2.3 - Determination of palaeodoses

22 The most commonly used approach in OSL dating consists of exploiting only part of the luminescence signal emitted in response to an optical stimulation, called the "fast" component. As its name indicates, this approach is favourable because of the rapid bleaching of this component when quartz grains are transported and deposited.

However, it suffers from the limited capacity of the used traps to accumulate the dose. Indeed, when the traps associated with this component are "full", the relation between the time and the radiation dose does not exist anymore and the dose saturation is reached; this phenomenon strongly limits the applicability of the OSL technique based on the fast component. In the present study, preliminary tests have revealed, for 14 samples among the 17 studied, a dose saturation of the fast component. Consequently, we turned toward a different OSL signal, obtained by thermal transfer (TT-OSL). The TT-OSL signal is associated with traps which have a higher capacity to accumulate radiation doses than the traps corresponding to the fast component, and then have a higher saturation dose.

23 In general, in luminescence dating, it is not possible to directly quantify the palaeodose. This is why the different processes carried out to determine this value consist of comparing natural luminescence signals (induced by past radiations) with luminescence signals produced by known radiation doses, delivered in the laboratory with a calibrated radioactive source.

24 For the three most recent samples datable by OSL in using the fast component of the signal (DOSL6b, ROSL10, ROSL11), the Single Aliquot Regenerative dose (SAR) protocol was applied (Murray and Wintle 2000). This protocol allows to measure all luminescence signals, whether induced by natural radiations or by artificial regeneration doses given in the laboratory, on a single fraction of sample, called "aliquot" (fig. 3). Practically, the protocol includes several cycles of measurements in which the only variable parameter is the regenerative dose. In addition, the OSL signal induced by a constant dose is measured during each cycle for evaluating sensitivity changes of the material and, if necessary, for correcting them. The effectiveness of this correction has been checked by applying twice the same regeneration dose (during the second and the last measurement cycles) and by comparing the normalized luminescence signals. The final step of the protocol consists of measuring a signal when no dose was previously given. This step allows to check for the absence of thermal transfer during the preheat operations preceding each OSL measurement.

25 From the generic SAR protocol just described, it was necessary to define the optimal parameters for each of the three samples, in particular the preheating conditions for removing the unstable components of the signal which are inappropriate for dating. A series of tests (dependence of the dose with preheating temperature, efficiency of the protocol to recover a known dose) have thus allowed to adapt the generic SAR protocol to each of these three samples (DOSL6b, ROSL10, ROSL11). Subsequently, 32 aliquots of each of these samples were measured. The obtained paleodoses were accepted when the criteria of applicability of the SAR protocol were validated. The dispersion of the palaeodose values obtained for each sample has been expressed as a percentage of overdispersion (OD). This value takes into account the dispersion of the results beyond the statistical "normal" variability. Thus, in an ideal situation in which each quartz grain would have been completely bleached at the time of deposition, and in which all the grains would have precisely received the same radiation dose, the OD percentage would be non-existent. In light of the really low OD percentages obtained for the OSL dated samples (3±1 % for DOSL6b, 6±1 % for ROSL10 and 2±1 % for ROSL11), we can conclude that these sediments have been properly bleached and that all the grains have been exposed to the same dose rate. Consequently, the central age model (Galbraith et

al. 1999), based on the calculation of an average of the individual palaeodoses, has been used for each of these three samples.

26 For all the other samples, for which the fast component of the quartz natural OSL signal was saturated, it was necessary to adopt a different protocol. An OSL signal obtained by thermal transfer (TT-OSL) has been exploited because of its very high saturation dose (Wang et al. 2006). Specifically, it has been measured in the following manner: a first preheat followed by OSL stimulation allowed to empty the "saturated" traps (the fast component of the signal). The sample was then heated once again, at 260°C during 10 s, in order to transfer the charges trapped in deeper traps (and capable of accumulating larger doses) towards the previously emptied traps : for this reason, this signal is called TT-OSL. A second optical stimulation thus allowed liberating the newly transferred charges.

27 Experiments carried out with a solar simulator (reproducing natural light) demonstrated that the bleaching decay of the TT-OSL signal is much lower than the fast component of the OSL (Hernandez 2011). When a few seconds of exposure to an intense light are enough to reduce the fast component of the OSL signal of quartz grains to an undetectable level, it is necessary to extend this duration by several days for the TT-OSL signal to reach its minimum residual (fig. 4). Indirectly, this characteristic makes the SAR protocol inapplicable when measuring the TT-OSL signal (because a full resetting by light is unachievable between cycles). Indeed, it becomes in this case necessary, at the end of each cycle, to heat the aliquot at a high temperature. Unfortunately, this heating induces important sensitivity changes which up to date can not be corrected for (Hernandez et al. 2012). In order to overcome this difficulty, a protocol including the measurement of only one regeneration dose per aliquot was set up. In this protocol, the dose growth curve was built from numerous aliquots previously bleached in a solar simulator. For each regeneration dose, four aliquots were measured, with the aim of taking into account the variability of the luminescence produced by the various quartz grains present in a given sample. In parallel, another group of aliquots, kept away from the light, have been used to measure the natural TT-OSL signals. Thus, in a similar manner to the "classical" SAR protocol, it was possible, by interpolating the intensities of the natural signals measured onto the average growth curve set up for each sample, to obtain as many palaeodose values as measured aliquots, that is to say 30 in the context of the present study (fig. 5).

28 The OD percentages calculated for each sample indicate that the dispersion of the palaeodose values is globally higher for the Romentères samples (between 11±1 % and 22±3 %) than for those from Duclos (between 6±1 % and 12±2 %). The reasons of this variability are numerous (heterogeneity of the alpha and beta dose rates, heterogeneous bleaching of the grains at the time of deposition) and are then difficult to assess precisely. However, the comparisons of the palaeodose values obtained with the OSL and TT-OSL signals for the ROSL11 sample bring to light some possible answers. The overdispersion percentage observed in the case of the OSL signal (2±1%) is much lower than for the TT-OSL signal (11±1 %). As the fast component of the OSL signal is bleached much more quickly than the TT-OSL signal, it could be suggested that the higher dispersion percentage of the TT-OSL indicates an incomplete bleaching of the grains, while the OSL signals would have been totally reset. Following this reasoning, the equivalent average dose determined with the TT-OSL signal would be overestimated in comparison to the OSL signal. But one can notice that it is not the case

(average equivalent dose of 65±0.5 Gy for the OSL signal and 63±2 Gy for the TT-OSL signal); consequently, a part of the dispersion of the equivalent doses measured with the TT-OSL signal is independent of the bleaching problems, at least for this sample. On the other hand, for the ROSL2, ROSL4 and ROSL6 samples, for which the dispersion of the results is particularly high, the question of bleaching remains open. However, the symmetry in the palaeodoses distribution (fig. 6) has led to treating these three samples in the same manner as the others, i.e. by calculating an age from the average palaeodose, without excluding any value.

Figure 3 - Description of SAR protocol as conventionally used for the determination of paleodose in OSL dating. Top: Table describing the sequence of operations performed during a cycle of the protocol. This is repeated as many times as regenerative doses are administered. The first cycle corresponds to a zero dose in order to measure the «natural»signal. Before each measurement of luminescence, it is necessary to preheat the sample to remove the trapped electron charges unsuitable for dating: whose life time is less than or of the order of magnitude of the expected age. A reset signal is carried out at the end of each cycle in order to prevent charges from accumulating from one cycle to another. These parameters must be adapted to the physical characteristics of each sample. Bottom: growth curve of the fast component OSL signal as a function of the regenerative dose obtained by applying the SAR protocol on an aliquot of the sample ROSL11. The value is obtained by interpolation of the «natural» signal on the growth curve. In this case, the value of the paleodose is approximately 63Gy.

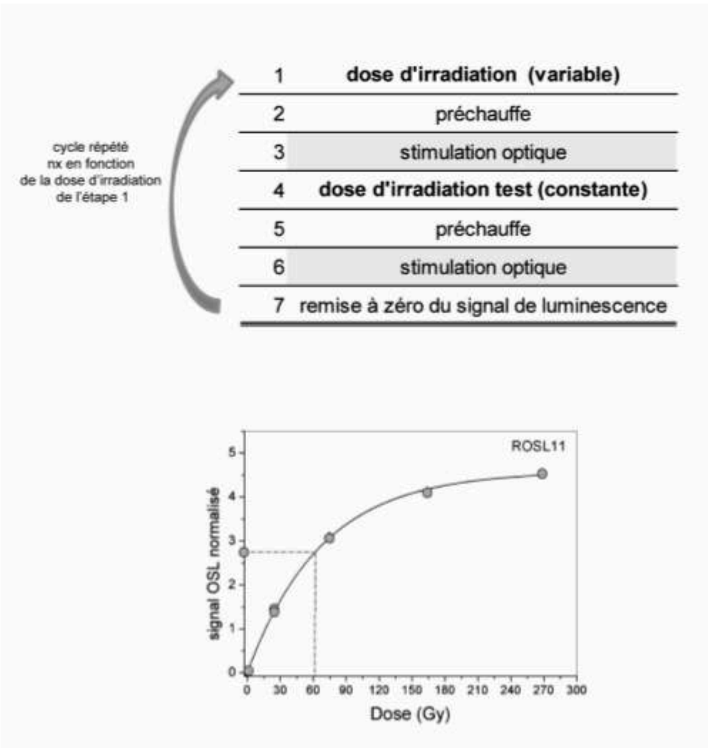


Figure 4 - Decay curves of the fast component of the OSL signal and TT-OSL signal as a function of time of exposure to a solar simulator (SOL500) for the sample DOSL2. After one minute of stimulation, the fast component of the OSL signal is fully bleached, while the intensity of the signal TT-OSL has reduced by only 20% from its maximum bleaching ability. We note that the TT-OSL signal can not be completely bleached but reaches a minimum residual level after about six days.

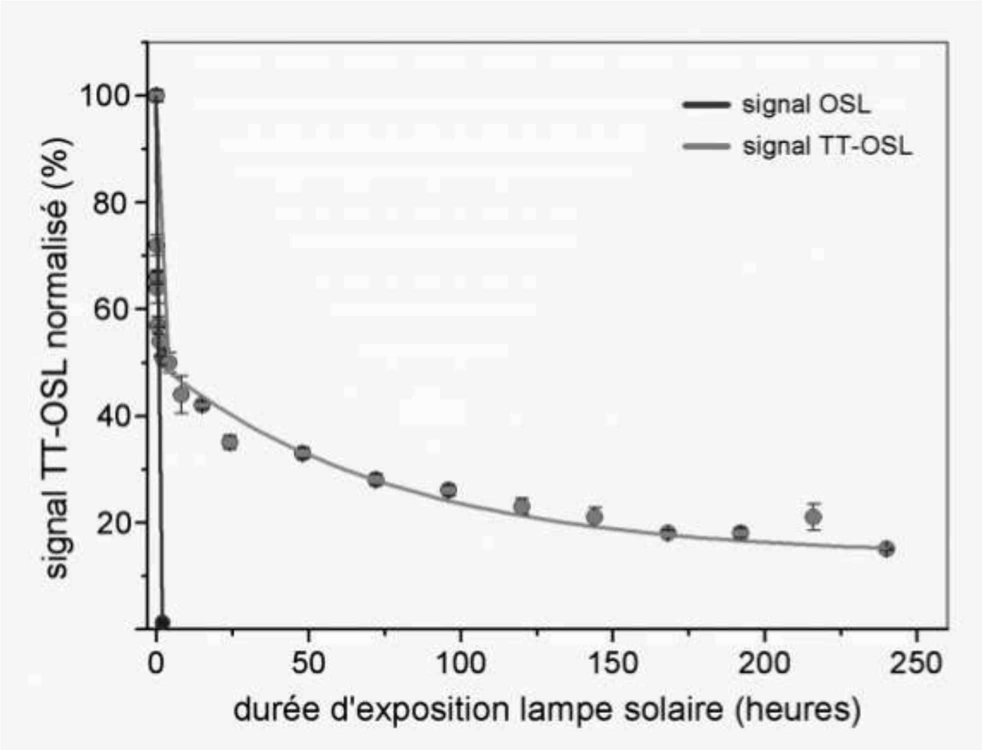


Figure 5 - Dose growth curve of the sample DOSL8 established from the measurement of TT-OSL signal. The error associated with each point of regenerative dose corresponds to the standard deviation of the four measurements performed on four different aliquots for each regenerative dose. The significant value of luminescence measured for a zero dose corresponds to the residue of TT-OSL signal remaining after bleaching (Hernandez 2011). Meanwhile, natural TT-OSL signals were measured from 30 aliquots of the sample. By interpolation of these values on the mean growth curv, 30 paleodoses were determined.

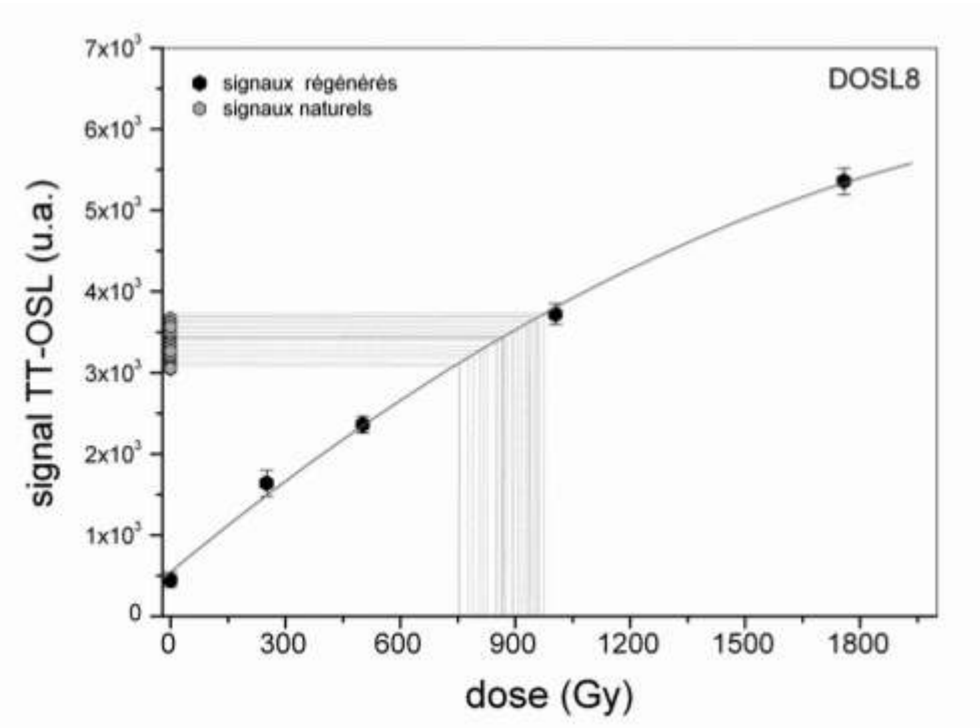
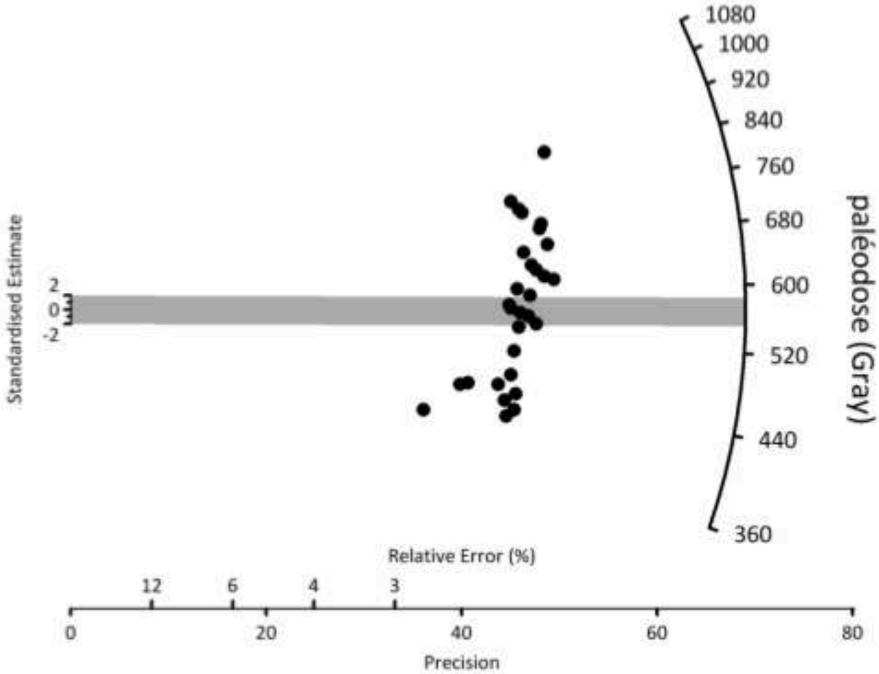


Figure 6 - Radial plot of 30 paleodose values of the sample ROSL6 obtained by TT-OSL in function of their statistical error. This sample corresponds to that provided the greater paleodose values dispersion (OD= 22±3 %). The distribution shows that the paleodoses values are distributed symmetrically around the mean value represented by the band gray (2 sigma).



3.3 - Dating of the heated rocks (TL)

3.3.1 - Determination of the palaeodose

29 Among the 66 artefacts made of flint and quartzite which showed signs of past heating, 54 were selected for TL measurements. As a first step, tests aiming to verify the efficiency of the archaeological heating to fully remove the previously accumulated doses, were carried out. They allowed to select only 4 samples : two flints and one quartzite from Romentères and one quartzite from Duclos. For these four samples, a series of preheating tests allowed to determine the optimum measurement parameters for removing the unstable components of the TL signal (a preheating at 290°C during 10 s was carried out for the two heated flints labelled BDX14684 and BDX14687, and at 320°C and 270°C during 10 s for the quartzite samples BDX12762 et BDX13225, respectively). The equivalent doses were determined following a protocol of added doses in using multiple aliquots aimed at defining the growth curve giving the variations of the TL intensity with the added dose (named first growth curve). Generally, the TL protocol includes the construction of a second growth curve from signals induced by regenerated doses after the sample powder has been heated at 350°C during 1h30 ; this second curve allows then to refine the palaeodose determination obtained by analysing the first growth curve. However, because of important sensitivity changes observed after the heating at 350°C, we were unable to construct the second growth curve and then no correction of the palaeodose could be made in the present study. Considering the relatively high palaeodose values of our samples (between 228±6

Gy and 340±18 Gy, see tabl. 2), this correction would have however not been significant (below 5 %).

Table 1 - Summary table of data involved on the age calculation of the sedimentary quartz samples from Duclos and Romentères. For the external alpha and beta dose rates, correcting factors have been applied, taking into account the size of the quartz grains used to the paleodose determination (20-41µm) from the values determined by Brennan et al. (1991) and Mejdhal (1979). In order to weight the alpha contribution in the total dose rate, a factor of 5µGy/10^3/cm^2 have been used. The percentage of overdispersion is noted OD in the table. The mean values of paleodoses, expressed in gray (Gy), were calculated using the Central Age Model (CAM) from Galbraith et al., 1999. Ages are given in kilo years and their uncertainties correspond to 1 sigma.

					Débits de dose (µGy/an)					paléodose			
éch.	prof. (m)	K (%)	U (ppm)	Th (ppm)	alpha	beta	gamma	cosmique	**Total**	méthode	OD (%)	**CAM (Gy)**	**Age (ka)**
DOSL6b	0.7	0.84 ± 0.02	3.53 ± 0.05	10.28 ± 0.13	241 ± 45	1189 ± 17	941 ± 9	209	2533 ± 49	OSL	3 ± 1	32.0 ± 0.2	**13± 1**
DOSL5	1.2	0.66 ± 0.02	3.17 ± 0.04	9.93 ± 0.12	200 ± 42	1027 ± 14	856 ± 7	213	2297 ± 45		12 ± 2	104 ± 2	**45 ± 3**
DOSL1	1.3	0.63 ± 0.02	3.35 ± 0.04	9.55 ± 0.12	201 ± 43	1017 ± 13	850 ± 7	206	2274 ± 45		6 ± 1	103 ± 1	**45 ± 3**
DOSL2	1.7	0.64 ± 0.02	3.56 ± 0.04	10.84 ± 0.11	221 ± 47	1073 ± 12	925 ± 7	213	2432 ± 49		10 ± 1	459 ± 11	**189 ± 13**
DOSL3	1.9	0.60 ± 0.02	2.99 ± 0.04	10.52 ± 0.12	200 ± 43	974 ± 14	848 ± 7	207	2231 ± 45	TT-OSL	9 ± 1	524 ± 17	**235 ± 17**
DOSL4	2.2	0.61 ± 0.02	3.05 ± 0.04	10.64 ± 0.13	203 ± 43	991 ± 14	862 ± 8	195	2252 ± 46		5 ± 1	616 ± 10	**274 ± 18**
DOSL7	2.5	0.74 ± 0.02	3.24 ± 0.04	11.80 ± 0.13	222 ± 47	1125 ± 14	956 ± 8	178	2481 ± 50		9 ± 1	633 ± 11	**255 ± 18**
DOSL8	2.9	0.85 ± 0.02	2.95 ± 0.04	12.10 ± 0.14	216 ± 46	1167 ± 16	962 ± 8	173	2517 ± 50		8 ± 1	877 ± 14	**348 ± 22**
ROSL10	0.46	0.63 ± 0.01	2.90 ± 0.03	8.15 ± 0.09	177 ± 38	951 ± 11	763 ± 6	226	2117 ± 40	OSL	6 ± 1	4.7 ± 0.1	**2.2 ± 0.1**
ROSL11	0.85	0.85 ± 0.02	3.26 ± 0.03	9.40 ± 0.10	202 ± 43	1171 ± 12	899 ± 6	231	2503 ± 45	OSL	2 ± 1	65.0 ± 0.5	**26 ± 2**
										TT-OSL	11 ± 1	63 ± 2	**25 ± 2**
ROSL1	1.08	0.70 ± 0.02	3.13 ± 0.04	8.95 ± 0.11	193 ± 41	1048 ± 14	836 ± 7	195	2272 ± 44		14 ± 2	143 ± 5	**63 ± 5**
ROSL2	1.24	0.79 ± 0.02	2.98 ± 0.04	9.59 ± 0.12	195 ± 41	1104 ± 16	867 ± 8	211	2378 ± 45		16 ± 2	181 ± 6	**76 ± 6**
ROSL3	1.46	0.72 ± 0.02	3.27 ± 0.04	9.43 ± 0.11	202 ± 43	1086 ± 13	873 ± 7	204	2366 ± 45		12 ± 2	231 ± 5	**98 ± 7**
ROSL4	1.71	0.80 ± 0.02	3.35 ± 0.05	9.78 ± 0.13	209 ± 44	1157 ± 17	913 ± 9	196	2474 ± 48	TT-OSL	16 ± 2	234 ± 8	**94± 7**
ROSL5	1.95	0.67 ± 0.02	3.48 ± 0.04	10.57 ± 0.12	221 ± 47	1104 ± 13	932 ± 7	186	2443 ± 49		13 ± 2	410 ± 11	**168± 12**
ROSL6	2.26	0.66 ± 0.01	3.56 ± 0.04	10.70 ± 0.10	225 ± 48	1108 ± 11	942 ± 6	209	2485± 49		22 ± 3	570 ± 25	**229± 18**
ROSL7	2.56	0.61 ± 0.01	3.30 ± 0.03	10.62 ± 0.10	216 ± 46	1043 ± 10	903 ± 6	198	2360 ± 48		14 ± 2	749 ± 20	**317± 23**

3.3.2 - Determination of the dose rate

Even though the internal dose of sedimentary quartz is considered as negligible because of their very low radioisotopic contents, it was necessary to determine this component for the flint and quartzite samples. The K, U and Th contents were determined for each sample by ICPMS at the CRPG-CNRS, Nancy, using a fraction of crushed rock. Ordinarily, in the preparation of rocks for TL dating, the external 2 mm of the artefact are eliminated in order to remove the external alpha and beta contributions. In the present study, the external layer of the samples from Romentères was sawed off. However, the morphology of the artefact from Duclos did not allow to perform this operation; consequently, the sample was crushed in its totality. The external alpha contribution was considered negligible given the low proportion of volume affected by these short distance radiations (20 µm). However, the external beta dose which is difficult to quantify experimentally cannot be ignored (the beta particle range is about 2 mm) and was then numerically computed by G. Guérin through the GEANT4 system (Guérin 2011). This dose contribution was taken into account in the age calculation of this sample. Finally, it has not been possible to determine the external dose rates from radiometric measurements in the immediate environment of the samples because of the rescue excavations. As mentionned previously, the gamma dose rates were estimated from the radiometric data obtained from the sediment samples taken for the OSL, and located as close as possible in stratigraphy to the dated artefacts.

Tableau 2 - Summary table of data involved in the age calculation of burnt rock samples from Duclos and Romentères. The efficiency coefficients of alpha particles (S-alpha, expressed in Gy for 10^3 alpha/cm^2) were determined experimentally. Ages are expressed in kilo years and their uncertainty is given at 1 sigma.

								débit de dose (µGy a^{-1})							
								interne		externe					
site	un. strati.	échantillon	nature	K (%)	U (ppm)	Th (ppm)	S-alpha	alpha	beta	cos m.	beta	gamma	**Total**	**paléodose (Gy)**	**Age (ka)**
Duclos	BT1	BDX12762	quartzite	0.06 ±0.02	1.97 ±0.05	7.25 ±0.2	5	301 ±64	66 ±1	213	65 ±3	856 ± 7	1501 ±64	254 ± 19	**169 ± 19**
Romentères	BT1	BDX14687	silex	0.05 ±0.02	0.91 ±0.05	0.43 ±0.2	12.1	186 ± 28	18 ± 1	204	0	873 ± 7	1282 ± 29	316 ± 26	**246 ± 22**
	BT2	BDX14684	silex	0.02 ±0.02	0.10 ±0.05	0.18 ±0.2	11.5	27 ± 13	4 ± 1	186	0	932 ± 7	1148 ± 15	340 ± 18	**296 ± 22**
		BDX13225	quartzite	0.17 ±0.02	1.04 ±0.05	3.36 ± 0.2	4.4	131 ± 7	38 ± 1	209	0	942 ± 6	1320 ± 10	228 ± 6	**172 ± 9**

4 - Chronostratigraphic results

31 The dates resulting from OSL and TT-OSL signals of sedimentary quartz have allowed to precise the chronostratigraphy of the Duclos and Romentères sequences, and has facilitated their comparison (fig. 7). For the “upper silts”, the ages are between 2.2±0.1 ky and 98±7 ky in Romentères and between 13±1 ky and 45±3 ky in Duclos. The most recent age (~2 ky) obtained for the Horizon E sample in Romentères has been interpreted as the result of the loess reworking at a recent time, during the Holocene. The “primary” loess (not mixed up with gravels brought in by colluviation) has yielded an age of 26±2 ky at Romentères and of 13±1 ky in Duclos i.e. MIS 2 (Upper Pleniglacial). This age is similar to the chronological data known for loess deposits in northern France (Antoine *et al.* 2003b). In Romentères, four dates were obtained on the underlying colluvial deposits: they yielded ages contemporary with MIS 3 and the second half of MIS 5. In Duclos, two dates for these colluvial deposits have allowed their attribution to MIS 3 (Middle Pleniglacial). These results indicate that the “Upper silts” deposition covers the whole of the last climatic cycle. The resulting dates also show that the formation of the IIBT palaeoluvisol is bracketted by ages of ~45 ky and ~189 ky in Duclos, and ~94 ky and ~168 ky in Romentères, confirming the hypothesis of an Eemian age l.

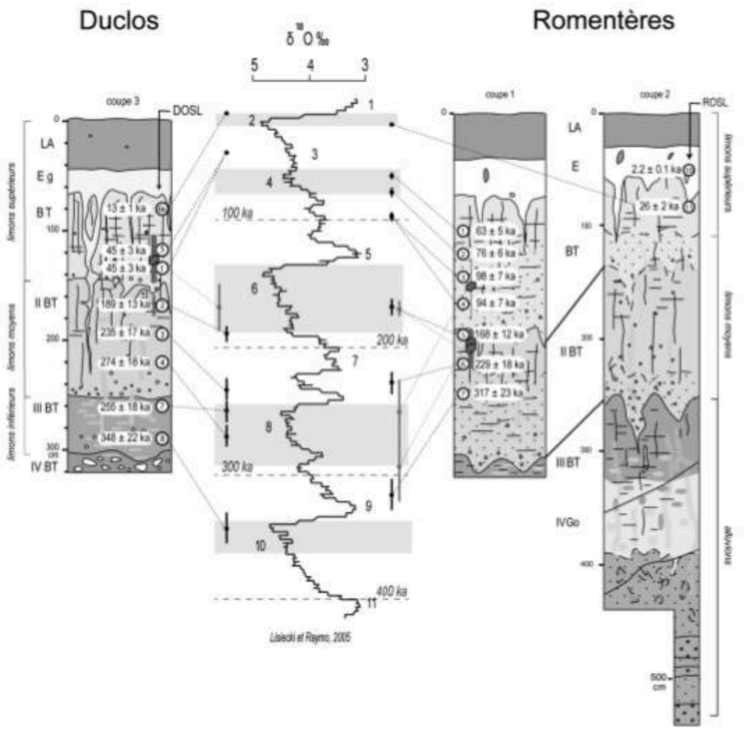
32 The resulting ages for the underlying unit’s samples are more difficult to match with the assumptions made from stratigraphic criteria. In Duclos, the formation of the IIBTg horizon is bracketted by four results : three come from the “middle silts” that overly the palaeosol (189±13 ky, 235±17 ky and 274±18 ky) and one from the “lower silts” affected by the palaeosol (255±18 ky). The attribution of the palaeosol to the intra-Saalian interglacial (MIS 7) is compatible with the resulting age for the “lower silts” (255±18 ka) and the more recent age obtained for the “middle silts” (189±13 ka) but it is incompatible with the two other results (235±17 ky and 274 ±18 ky). In Romentères, the “middle silts” have yielded ages between 168±12 ky and 317±23 ky, suggesting they were deposited over two climatic cycles. In Duclos, a long chronology stretching also across two climatic cycles is suggested by the dates from the lower silts (255±18 to 348±22 ky), which contradicts geological interpretations. Up to date, it has not been possible to understand the reason of these discrepancies.

33 The comparison of the OSL and TT-OSL dates with the TL dates provides additionnal informations, in spite of the small number of heated samples, on the taphonomic history of the lithic assemblages of each site. In Duclos, the industry is mostly found in deposits dated from the last cycle (MIS 5 to 3) and in part in levels attributed to the limit between MIS 7-6. The only sample dated by TL comes from the upper silts and

yielded an age of 169±19 ky (MIS 6), which is older than the age of the enclosing sediment (MIS 3), and confirms that part of the industry is found in a secondary geological position. The hypothesis that the remains come from a single assemblage, a witness of an occupation contemporary of MIS 6 or, at the most, on the limit MIS 7-6, can be put forward. According to this hypothesis, the lithic artefacts from the middle silts would thus geologically be in place.

34 By comparison, at Romentères, the situation is the opposite. The main series is found in silty colluvial deposits (middle silts) dated between MIS 7 and 6. Part of the assemblage, smaller in number, is found in sediments dated to the MIS 5 (upper silts). Although one of the resulting TL dates perfectly agrees with the age of the associated sediments (MIS 6), the two other results on heated flints seem to indicate the presence of older artefacts within the assemblage; these lithic artefacts would thus correspond to one or several occupations contemporary with MIS 8 and 9. As a hypothesis, these old ages could be associated with a small series of Acheulean appearances.

Figure 7 - Correlation between the dating results and the marine isotopic stages. The location of samples is represented by circles for the sedimentary quartz and by hexagons for burnt rocks. Archaeological levels are represented by gray lines more or less thick depending on the density of material excavated.



5 - CONCLUSIONS

35 Since its advent, the OSL dating method has become a powerful tool for dating sedimentary deposits. However, it has seldom been applied to the study of Lower and Lower-Middle Palaeolithic sites due to its limited chronological range (when the OSL fast component is used for the age determination). From a methodological point of view, the present study allowed to show the interest and the reliability of an approach

using the TT-OSL signal for dating sedimentary quartz of Upper and Middle Pleistocene age.

36 Through this new methodology, the resulting ages have confirmed the stratigraphic recognition of the Eemian palaeosol and strengthened the understanding of sedimentation mechanisms during the last glacial cycle in the Aquitaine region (Bertran *et al.* 2011). However, the discrepancy between certain dates and some hypotheses based on the geomorphologic data related to Middle Pleistocene deposits shows the necessity to carry on with these kind of studies in order to resolve the contradiction of the currently available results.

37 Despite the small number of samples which could be dated by TL, new chronological markers are now available for the Middle Pleistocene industries of the Aquitaine region. If the attribution to MIS 6 of the Early Middle Palaeolithic with rare bifaces at Romentères fits well with the chronological framework accepted for this type of industry in the northern part of the Aquitaine Basin (i.e. Brenet *et al.* 2008), the resulting dates for the Acheulean of Iberian type (or Pyrenees-Garonne Acheulean) are unprecedented. The latter has long been considered as strictly predating the manifestations of the Middle Palaeolithic in this region. Nonetheless, the attribution to the beginning of MIS 6 of the industry found at Duclos, and potentially to MIS 8-9 of the Acheulean series at Romentères, tends to contradict this hypothesis and forces consideration of long contemporaneousness of these different technocomplexes in the same territory. This new perspective leads to consider their relation from the point of view of technical traditions and/ or exchanges.

This research has been done in the context of a doctoral thesis funded by the Aquitaine Region. We would like to thank Guillaume Guérin for doing the numerical simulations for the sample from Duclos.

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ABSTRACTS

The recent development work of the A65 highway has given the opportunity to broaden the corpus of Lower and Middle Palaeolithic open-air sites known in the southern Aquitaine basin. The sites of Duclos (Auriac, Pyrénées-Orientales) and Romentères (Le Vignau, Landes), discovered in this context, have yielded an abundant lithic record which has been attributed to the Acheulean of Iberian type for the earlier and, essentially, to the Early Middle Palaeolithic for the latter. The archaeological levels are intercalated in sequences that comprise aeolian silts and colluvial units separated by interglacial luvisols. The pedostratigraphic context of the two sites has provided a rare opportunity to propose a reliable chronostratigraphic framework in this area. To achieve this objective, a study combining several methods of luminescence dating was conducted to complete the geomorphological data. Optically Stimulated Luminescence (OSL) and Thermically Transferred OSL (TT-OSL) dating methods were applied in order to determine the ages of sedimentary quartz grains and thus, of the sampled deposits. In parallel, heated flint and quartzite samples were dated by thermoluminescence (TL). The Pyrenees-Garonne Acheulean industry of Duclos has been attributed to the boundary between Marine Isotopic Stages (MIS) 7 and 6 while the human occupations of Romentères date from MIS 6 for the most recent series (Early Middle Palaeolithic) and from MIS 9 and 8 for the older.

INDEX

Keywords: Acheulean, Early Middle Palaeolithic, Pyrenees-Garonne region, Middle Pleistocene, chronostratigraphical framework, luminescence dating.

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