Close encounters of the third kind? Neanderthals and modern humans in Belgium, a bone story
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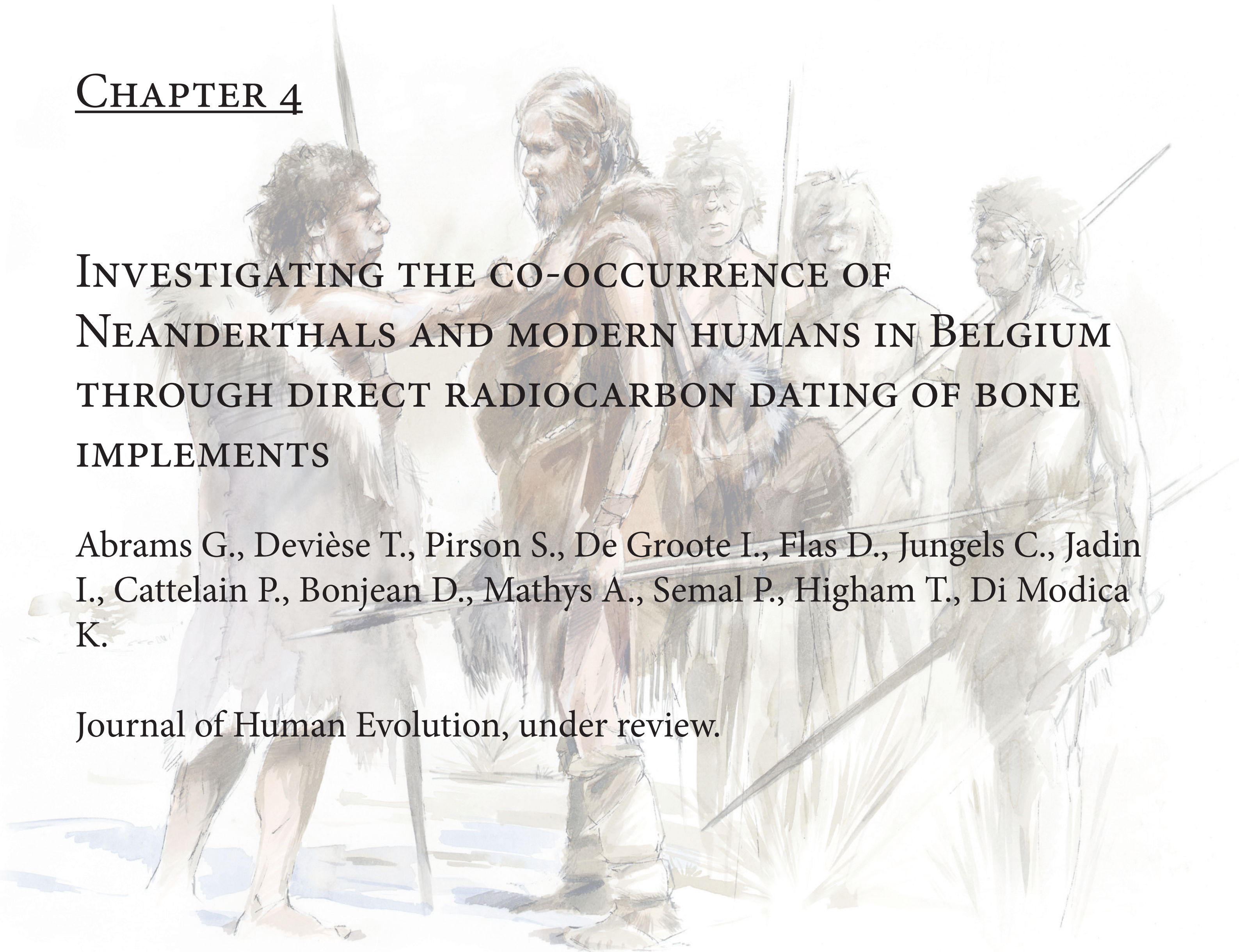

Chapter 4

Investigating the co-occurrence of Neanderthals and modern humans in Belgium through direct radiocarbon dating of bone implements

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Contribution of G. Abrams: coordination of the dating research programme, selection of samples, analysis and interpretation of data, writing of the manuscript.

ABSTRACT

Determining the timing of the transition between Neanderthals and anatomically modern humans (AMHs) is crucial in archaeology and paleoanthropology. While there is increasing evidence of admixture and co-existence of the two hominin species in Central and Eastern Europe, Belgium might show a different scenario. Recent ancient DNA and radiocarbon analyses seem to indicate a hiatus in the occupation of the territory. However, this interpretation is based on a limited number of hominin specimens because of their scarcity in the archaeological record. Mousterian and Aurignacian industries, associated with Neanderthals and AMHs respectively, are present in much larger quantities, and can also contribute to define the timing of both occupations. Few radiocarbon dates, measured on ultrafiltered collagen, have been produced for these industries. These data show a possible coexistence of Mousterian (42,300–39,900 cal BP) and Aurignacian (41,650–39,250 cal BP). We revaluate here the chronology of the latest Mousterian and earliest Aurignacian cultural evidence using the compound specific radiocarbon dating approach which is the most robust pre-treatment method. Our new data obtained on diagnostic bone implements show that the latest Mousterian occurrence possibly ended around 45,900–42,900 cal BP (95% probability) and that the earliest Aurignacian started around 42,100–40,300 cal BP (95% probability) - a date that is much older than the dates previously obtained on the same objects. Also considering the dates on Lincombian-Ranisian-Jerzmanowician industries, this new data tends to confirm that there may have been a hiatus implying that Neanderthals and AMHs did not co-exist in this region.

1. Introduction

The dynamics of the transition between Late Neanderthals and early anatomically modern humans (AMHs) is the subject of intense debate: the location and duration of the coexistence of the two human populations. As well as their relation and the cultural exchanges that could have occurred during this transitional period, referred as the Middle Palaeolithic to Upper Palaeolithic Transition (MUPT), is still being discussed (d'Errico, 2003; Hublin, 2015; Soressi and Roussel, 2014). The precise chronological position of the different cultural facies, as well as the human remains associated with them, are therefore key elements that delineate the chronological framework within which Neanderthals and AMHs could have interacted. It bears upon the dynamics of colonization of Eurasia and the replacement of the last Neanderthals by AMHs. While evidence of interbreeding between Neanderthals, Denisovans and/or AMHs have been documented elsewhere (Fu et al., 2015; Hajdinjak et al., 2021; Massilani et al., 2020; Prüfer et al., 2021; Slon et al., 2018), Northern and Western Europe seem to have been populated only by Neanderthals until around 45,000–43,000 cal BP (Devièse et al., 2021; Hublin, 2015; Nigst et al., 2014). Moreover, genetic analyses show the absence of genetic flow from early AMH to late Neanderthal populations (Hajdinjak et al., 2018) as well as the absence of Neanderthal genes in Northern European Early Upper Palaeolithic modern humans (Posth et al., 2016). However, this interpretation is based on a limited number of hominin specimens because of their scarcity in the archaeological record. Mousterian and Aurignacian industries, associated with Neanderthals and AMHs respectively, are present in much larger quantities, and can also be used to define the timing of both occupations.

Prehistoric research in Belgium has yielded numerous sites attributed to the Mousterian (Di Modica, 2010; Di Modica et al., 2016; Ulrix-Closset, 1975), the Aurignacian (Dinnis and Flas, 2016; Otte, 1979), as well as two occurrences related to the Lincombian-Ranisian-Jerzmanowician (LRJ) transitional technocomplex (Flas, 2011b). However, most of these historical collections lack reliable contextual data (Abrams, 2018; Di Modica et al., 2016; Pirson et al., 2012). Before the present study, the most recent dates for the Late Mousterian industries in Belgium came from recent excavations at Scladina and Walou caves and suggested that the Mousterian

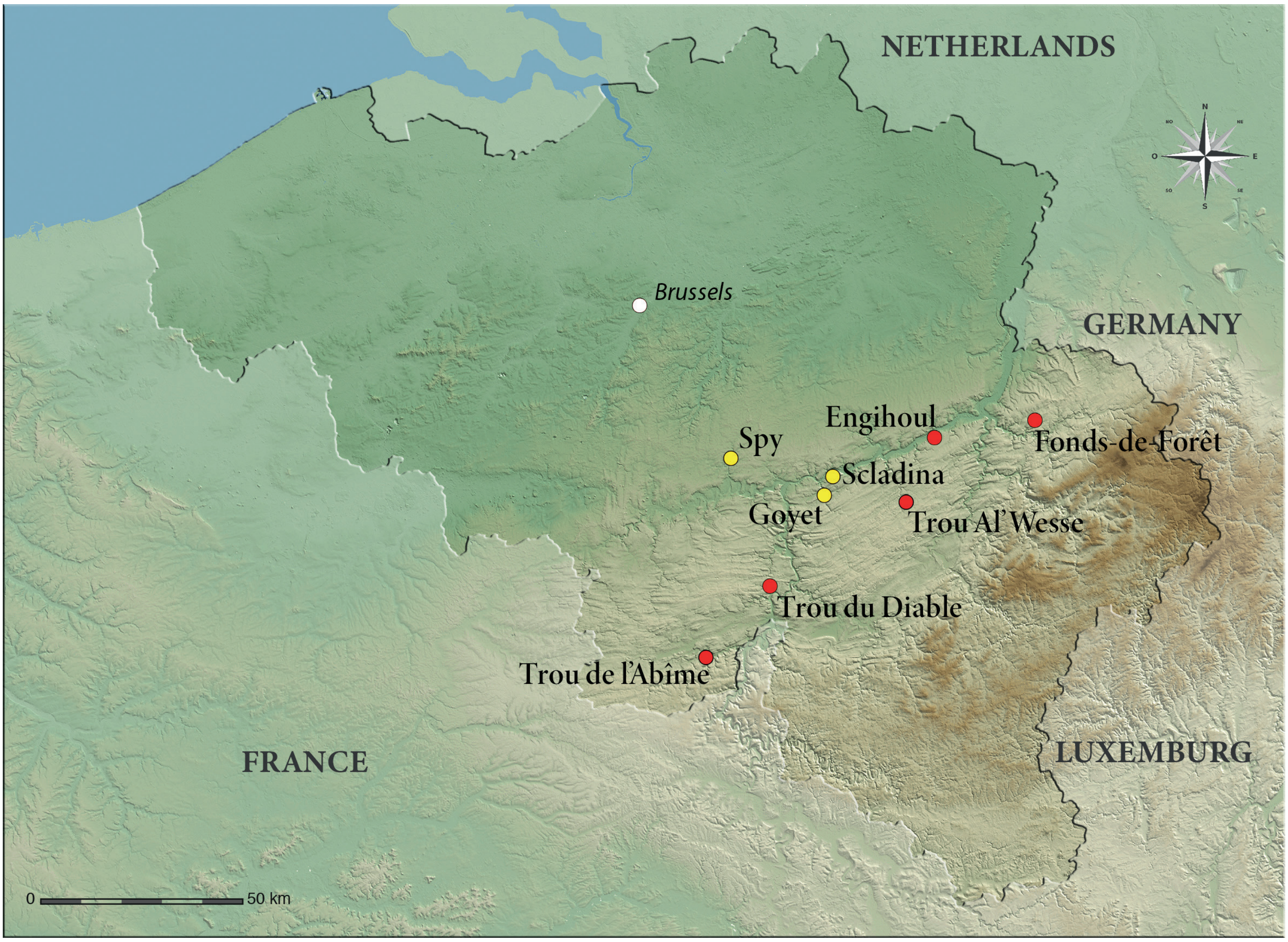

FIGURE 1. Map of Belgium showing the location of the sites where bone tools dated in this paper were found. Sites related to the dating of the Early Aurignacian are indicated in yellow and sites related to the dating of the Mousterian are figured in red.

ended around 42,000 cal BP (Fig. 1). Unfortunately, the dates were not produced on anthropogenically modified bones, which date human presence unequivocally compared to unmodified bones from the archaeological contexts which rely on our interpretation of sedimentary processes (Pirson et al., 2012). Conversely, the dating of the oldest Aurignacian occurrence in Belgium, was established on a spear point from Spy Cave dated to 38,100–36,500 cal BP (GrA-32619). This suggested a 4000-year hiatus between the Mousterian and Aurignacian. However, given the CN ratio of the bone point from Spy (3.61), this date should be considered as a minimum age (Dinnis and Flas, 2016; Flas et al., 2013; Pirson et al., 2012). Radiocarbon dates obtained by Semal and colleagues (2009) on collagen of the Spy Neanderthals partially filled this 4000-year hiatus (Semal et al., 2009). The survival of Late Neanderthals beyond the Mousterian allowed their hypothetical association with the LRJ, for which archaeological evidence was found in the historical archaeological record (Flas, 2011b; Semal et al., 2009). Based on this available data, the proposed scenario for the MUPT in Belgium was that the Mousterian ended around 42,000 cal BP, was followed by the LRJ (≈40,000 cal BP) and that the Aurignacian arrived at around 38,000 cal BP.

To refine the chronology of the transition between the Mousterian and the Aurignacian cultures and to discuss the occupation of North-western Europe during this period of cultural and human transition, we first selected human remains (Devièse et al., 2021) and then exclusively modified bones, which constitute, along with human remains, the best material to directly date human activities. Even though the use of bone retouchers persists without major modifications throughout the entire Palaeolithic (Jéquier et al., 2018; Tartar, 2012; Toniato et al., 2018), the use of hard animal materials was witness to profound changes during the MUPT, highlighted by the production of ornaments (Arrighi et al., 2019; Caron et al., 2011; d'Errico, 2003) and

Cultural attribution	Site	Individual	Description	Species	ID	P code	Conventional 14C Ages	Calibrated Ages (95.4 %)
Early Aurignacian	Goyet Cave	IG-11735	Spear point	Reindeer (?)	OxA-X-2767-15	HYP	34,000 ± 450 B.P.	40,200-37,500 cal B.P.
(Early Aurignacian)	Scladina Cave	Sc83-312-1	Fractured bone	Horse	OxA-34045*	AF	35,300 ± 650 B.P.	41,650-39,250 cal B.P.
					OxA-X-2762-10	HYP	34,300 ± 800 B.P.	41,100-37,200 cal B.P.
		Sc2007-140-1	Bone retoucher	Horse	OxA-34044*	AF	35,350 ± 650 B.P.	41,700-39,300 cal B.P.
					OxA-38390	HYP	36,400 ± 1,600 B.P.	44,300-38,200 cal B.P.
Early Aurignacian	Spy Cave	Spy SP2 - Spy 1954	Spear point	Reindeer	GrA-32619	/	32,830 ± 200 B.P.	38,100-36,500 cal B.P.
					OxA-X-2767-14	HYP	36,200 ± 550 B.P.	42,100-40,300 cal B.P.
Late Mousterian	Trou du Diable	TDD_RET40	Bone retoucher	Bison	OxA-X-2762-26	HYP	41,500 ± 1,800 B.P.	49,800-42,300 cal B.P.
		TDD_RET64	Bone retoucher	Cervid	OxA-X-2767-16	HYP	41,700 ± 1,200 B.P.	47,100-42,800 cal B.P.
		TDD_RET32	Bone retoucher	Reindeer	OxA-34222*	AF	36,250 ± 800 B.P.	42,300-39,900 cal B.P.
					OxA-X-2767-08	HYP	42,300 ± 1,300 B.P.	48,000-43,000 cal B.P.
		TDD_RET62	Bone retoucher	Horse	OxA-34223*	AF	36,500 ± 800 B.P.	42,400-40,100 cal B.P.
					OxA-X-2762-11	HYP	44,100 ± 3,000 B.P.	43,900 cal B.P.>
	Fonds-de-Forêt Cave	FDF_RET02	Bone retoucher	Horse	OxA-37797	HYP	41,800 ± 1,400 B.P.	48,000-42,700 cal B.P.
					OxA-37796	HYP	42,900 ± 1,600 B.P.	50,300-42,900 cal B.P.
		FDF_RET01	Bone retoucher	Deer	OxA-37773	HYP	42,500 ± 1,400 B.P.	48,700-42,900 cal B.P.
	Trou Al'Wesse	TAW545_RET01	Bone retoucher	Cervid	OxA-34219*	AF	45,700 ± 2,400 B.P.	53,000-45,300 cal B.P.
					OxA-38323	HYP	43,800 ± 2,100 B.P.	52,600-43,300 cal B.P.
		TAW545_RET03	Bone retoucher	Reindeer	OxA-34220*	AF	41,300 ± 1,400 B.P.	47,400-42,500 cal B.P.
					OxA-38392	HYP	45,100 ± 5,100 B.P.	43,800 cal B.P.>
	Engihoul	ENGIHOUL_RET26	Bone retoucher	Bear	OxA-38393	HYP	46,900 B.P. >	
	Trou de l'Abîme	CTA H7 67	Bone retoucher	Horse	OxA-34120*	AF	43,600 ± 1,900 B.P.	52,500-43,100 cal B.P.

TABLE 1. List of the modified bones dated in this study. PCode refers to pretreatment code; AF is ultrafiltered collagen and HYP denotes the extraction of hydroxyproline from hydrolysed bone collagen. The ID with "*" are unpublished dates obtained on ultrafiltered collagen reported in this study. All technical data is reported in Tables S1 and S2. The calibrated ages were obtained using OxCal 4.5 (Bronk Ramsey, 2009) and the IntCal20 calibration curve (Reimer et al., 2020).

weaponry, such as the bone points (Kitagawa and Conard, 2020; Tartar and White, 2013).

Not only the choice of the material to radiocarbon date is important, but also the method to prepare the samples before measurement. This is illustrated, for example, by our recent radiocarbon dating study on Belgian Neanderthals including those from Spy, Engis and Fonds-de-Forêt. Using the compound specific radiocarbon analysis (CSRA) approach targeting the amino acid hydroxyproline, we demonstrated that previous dates produced on collagen for the Neanderthal specimens from Spy were inaccurately young by up to 10,000 years due to the presence of unremoved contamination including conservation materials (Devièse et al., 2021).

In this study, we report a series of unpublished radiocarbon dates on bone tools that were obtained on collagen (Table 1). We also report a new set of dates on modified bones obtained using the CSRA approach. Some of the artifacts were cross dated using both pre-treatment methods (collagen and hydroxyproline), which allows us to discuss the reliability of all the results obtained on the bone artefacts and discuss the co-existence of Neanderthals and AMHs in North-western Europe.

2. Material and methods

2.1. Material

The reassessment of the collections and the ongoing field research in certain sites, e.g., Scladina Cave, gave us access to first-hand material to refine the chronology of the MUPT in Belgium. Most of the bone implements that were radiocarbon dated in this study were identified from the faunal debris collected during the excavations (Abrams, 2018; Flas et al., 2013). They have therefore avoided any consolidation treatment.
The bone points are testimony of an important diversification of the bone tools at the beginning of the Upper Palaeolithic (Flas et al., 2013; Jéquier, 2016; Kitagawa and Conard, 2020; Szmidt et al., 2010; Tartar and White, 2013). Because of their manufacture and aesthetic quality, typologically characteristic bone artifacts such as bone points (Figs. 2-3) were quickly identified and isolated during excavations, even by the pioneers operating in the 19th Century. The conservation history of these pieces was not always recorded, and it cannot therefore be excluded that animal glues, of the same type as those identified on Neanderthal scapula from Spy (Spy 772a; Devièse et al., 2021), were also used. Due to this possible conservative background, as well as their fragility and their cultural interest, it was not appropriate to sample them for dating. However, a split-base point from Goyet (Fig. 2) collected during the excavations led by the Royal Belgian Institute of Natural Sciences (RBINS) in 1938 and another fragment from Spy Cave (SOM Fig. S2) collected by the RBINS in 1954 have been dated in this study. The one from Spy Cave was discovered within the faunal material and previously dated to 38,100–36,500 cal BP (GrA-32619; Flas et al., 2013).

FIGURE 2. Split-base point from Goyet Cave (IG 11735). Image G. Abrams (©RBINS).

FIGURE 3. Bone point fragment from Spy Cave (Spy SP2 - Spy1954). Pictures C. Jungels; Image G. Abrams (©RBINS).

The intrinsic features of the bone retouchers do not allow a cultural attribution to be proposed with certainty. This is only possible in certain cases (Castel et al., 2003; Tartar, 2012), which are not documented in Belgium. The cultural attribution of these artefacts has been made possible, for recent and ongoing excavations, by stratigraphic observations and their association with lithic industries. The bone retoucher made from a horse metacarpal excavated in Scladina (Sc2007-140-1; Fig. 4A) is stratigraphically associated, in Unit T-RO, with an Upper Palaeolithic endscraper on a blade. This makes it possible to hypothesize its association with the Aurignacian, based on the radiocarbon results, as well as the other fragment of horse metacarpal bearing percussion notches (Sc83-312-1; Fig. 4D). These two bones, which seem to belong to a unique fragmented metacarpal, have been cross dated (AF and HYP). Other bone retouchers from the sites of Trou Al'Wesse (Fig. 5) and Trou de l'Abîme (Fig. 6) were excavated recently and are stratigraphically related to the Mousterian.

The cultural attribution of the bone retouchers recovered in the historic collections is much more challenging and we must remain cautious while we interpret this material. Stratigraphic data, when they exist, are much less precise and often subject to doubt. The reassessment of the Trou du Diable collection provided around 300 bone retouchers (Fig. 7; Abrams, 2018). Research, still in progress, has made it possible to attribute retouchers with certainty to the Mousterian period thanks to the identification of the lithic chips still embedded in the bone matrix whose origin comes from the use of these retouchers (Abrams, in press; Abrams et al, in progress). The archaeological material that was excavated in the 3rd Ossiferous Unit ('3e niveau ossifère') of Fonds-de-Forêt Cave, including the bone retouchers that were dated in this study (Fig. 8), is related to the Mousterian industry close to the one unearthed in La Quina (France; Ulrix-Closset, 1975). According to different scholars, the 3rd Ossiferous Unit was covered by other layers that yielded artifacts from Upper Palaeolithic such as Aurignacian, Upper Perigordian and Ahrensbourgian (Dewez, 1987; Otte, 1979). Bone artifacts from Gisement Paléolithique of Engihoul

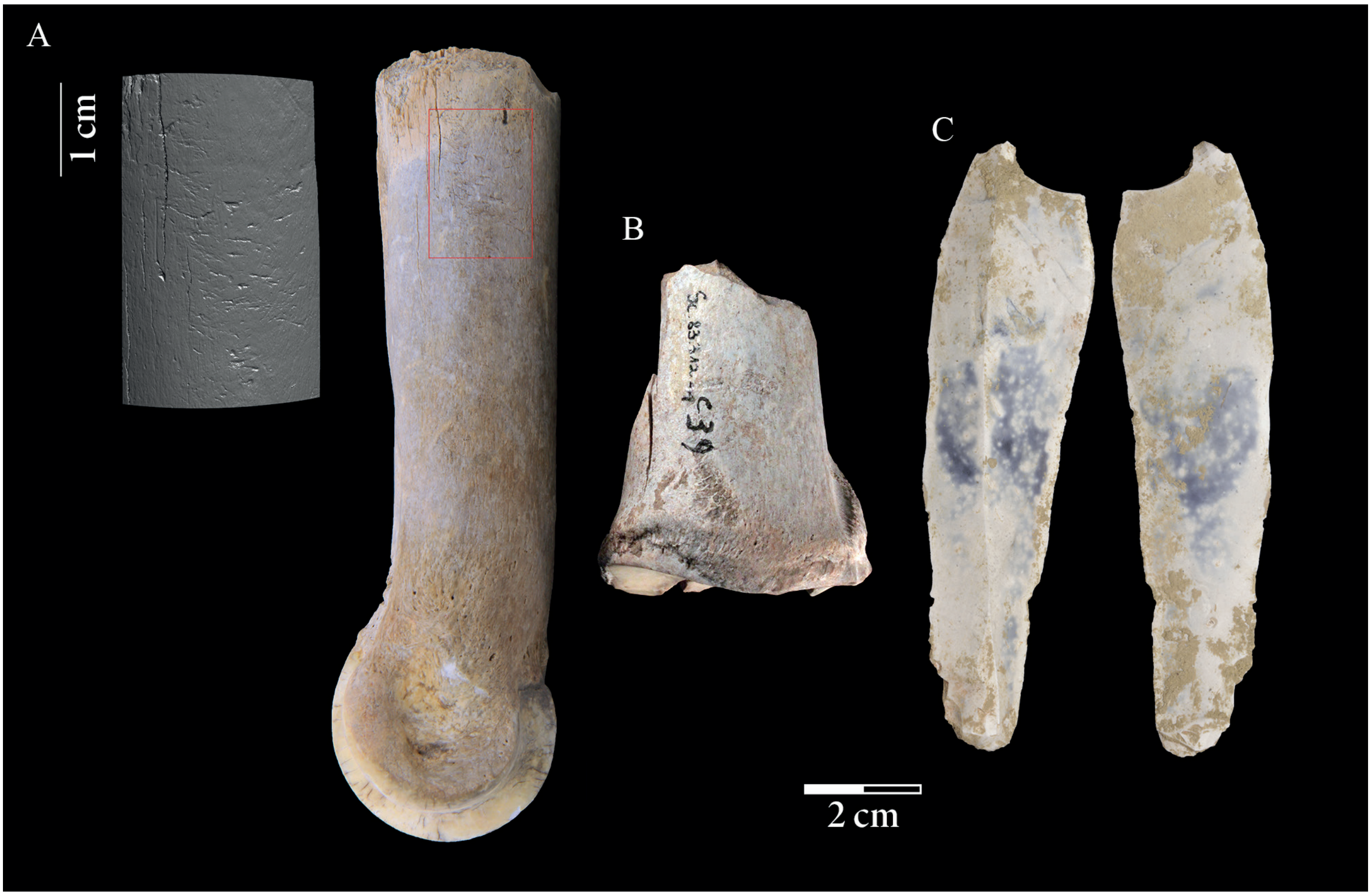

FIGURE 4. Tools attributed to the Aurignacian from Scladina Cave: (A) Bone retoucher made from a horse metacarpal (Sc2007-140-1); (B) 3D scan of the use surface; (C) Fractured proximal extremity from a horse metacarpal (Sc83-312-1) associated with the bone retoucher; (D) endscraper made on blade (SC2017-10-1). Image G. Abrams (©EMA).

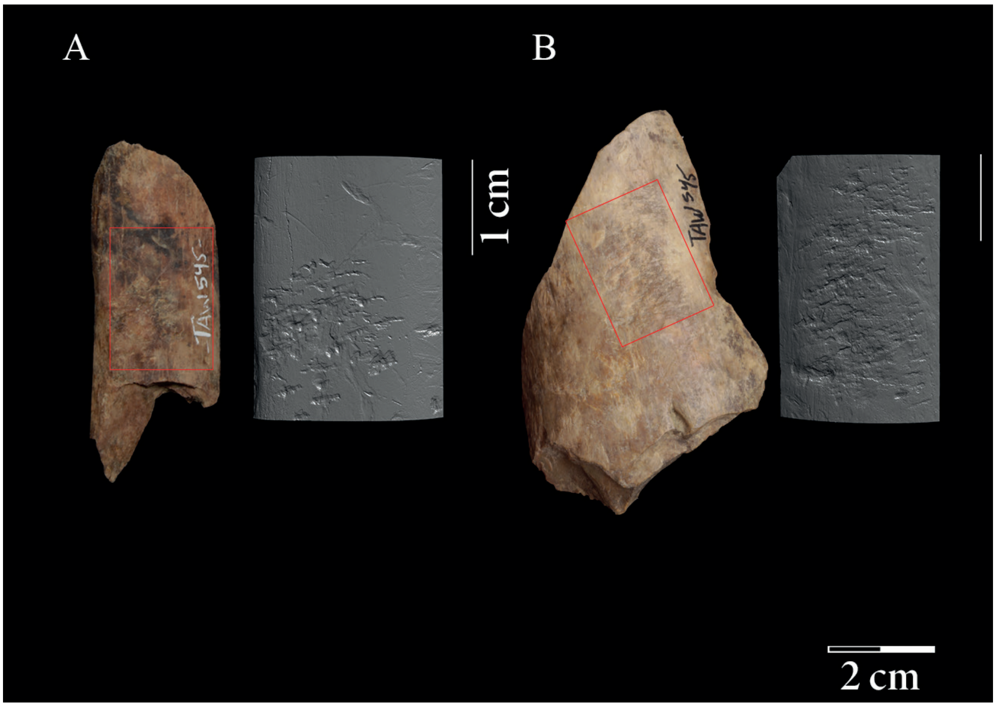

FIGURE 5. Bone retouchers associated with the Mousterian from Trou Al'Wesse: (A) TAW545_Ret01; C) TAW_Ret03; (B & D) 3D scan of the use surface. Image G. Abrams (©Préhistomuseum).

are much more challenging to interpret (Otte, 1979; Ulrix-Closset, 1975). Therefore, the cultural attribution (Mousterian) of the bone retoucher from Engihoul (Fig. 9) will be mainly based in this study on our radiocarbon results.

2.2. Methods

We (re)dated the specimens using the single amino acid radiocarbon dating method optimized at the Oxford Radiocarbon Accelerator Unit (ORAU), University of Oxford (Devièse et al., 2018a). This method involves separation of the underivatized amino acids from hydrolysed bone or tooth collagen samples using preparative Liquid Chromatography (Prep-LC). The amino acid hydroxyproline is isolated, combusted, graphitized and then measured on the AMS. This pre-treatment approach (Coded 'HYP' in the ORAU) is the most able technique to remove contaminants including, but not limited to, conservation materials (unless collagen-based glue has been applied). This is because HYP is a virtual biomarker for mammalian collagen and its successful extraction and radiocarbon measurement evidence a compound-specific measurement which necessarily eliminates all interfering contaminants. For these reasons single compound AMS dates are to be relied upon more than equivalent ages derived from bulk collagen, which can harbor unremoved contaminants derived through chemical cross-linking post-depositionally or from later treatment in museums or conservation laboratories (Higham, 2019). Our recent work suggests that one area of concern for underestimated radiocarbon ages on bulk collagen is in open air sites (eg; Bourrillon et al., 2018; Dinnis et al., 2019). We have hypothesized that this could be due to the greater presence of humic contaminants mobilized in aqueous solutions moving

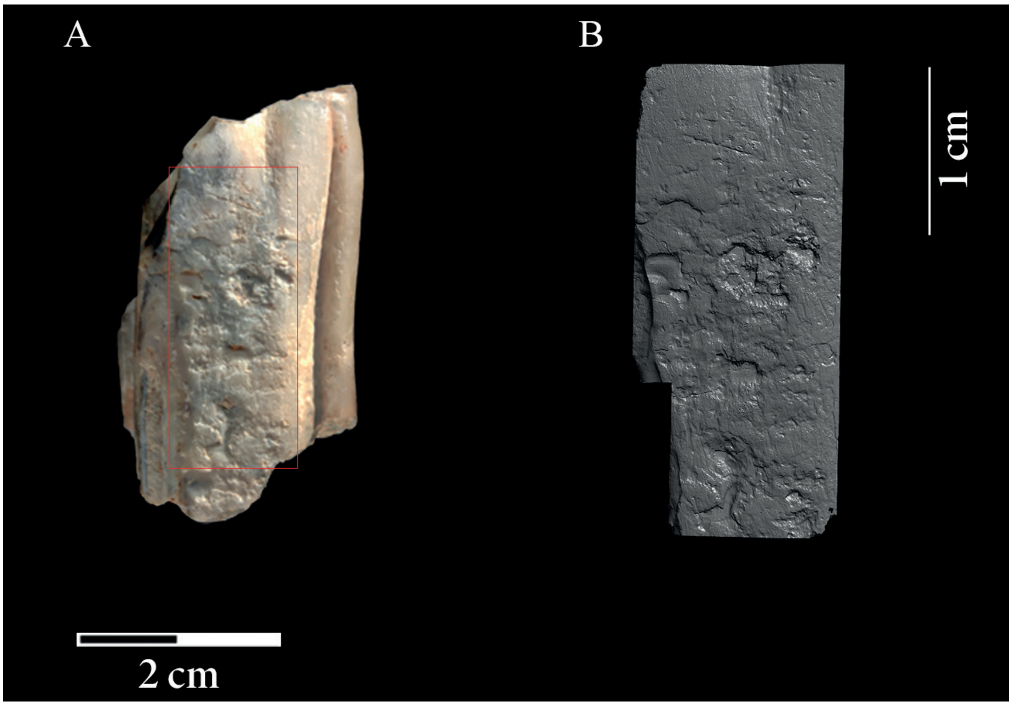

FIGURE 6. Bone retoucher (CTA H7 67) associated with the Mousterian from Trou de l'Abîme radiocarbon dated in this study. (B) 3D scan of the use surface. Image G. Abrams (©Cedarc-MMT).

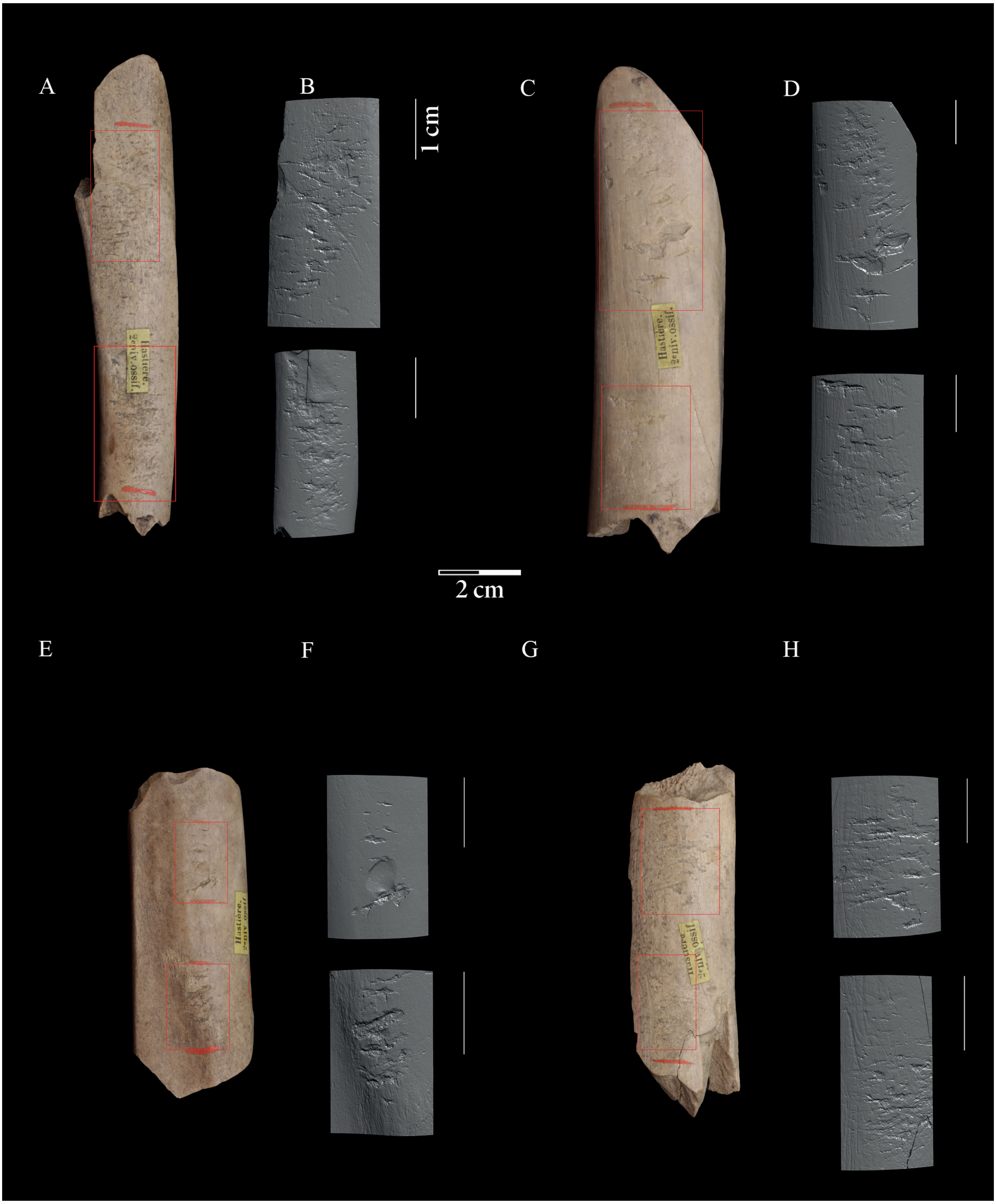

FIGURE 7. Bone retouchers from Trou du Diable, associated with the Mousterian radiocarbon dated in this study. (A) TDD_Ret40; (C) TDD_Ret64; (E) TDD_Ret32; (G) TDD_Ret62; (B, D, F, H) 3D scan of the use surfaces. Image G. Abrams (©RBINS).

down soil profiles more regularly than in deeper cave contexts. Previous workers have noted that there seem to be greater contamination effects on the outer apron deposits of cave sites compared with inside the dripline of the cave (Richter, 2002). This is an active area of research for our group currently.

The %C, %N and atomic C/N ratio were measured using an automated carbon and nitrogen elemental analyser (Carlo Erba EA1108) coupled with a continuous-flow isotope monitoring mass spectrometer (Europa Geo 20/20). Stable isotope ratios of carbon and nitrogen are reported in 'per mille' relative to VPDB and AIR with

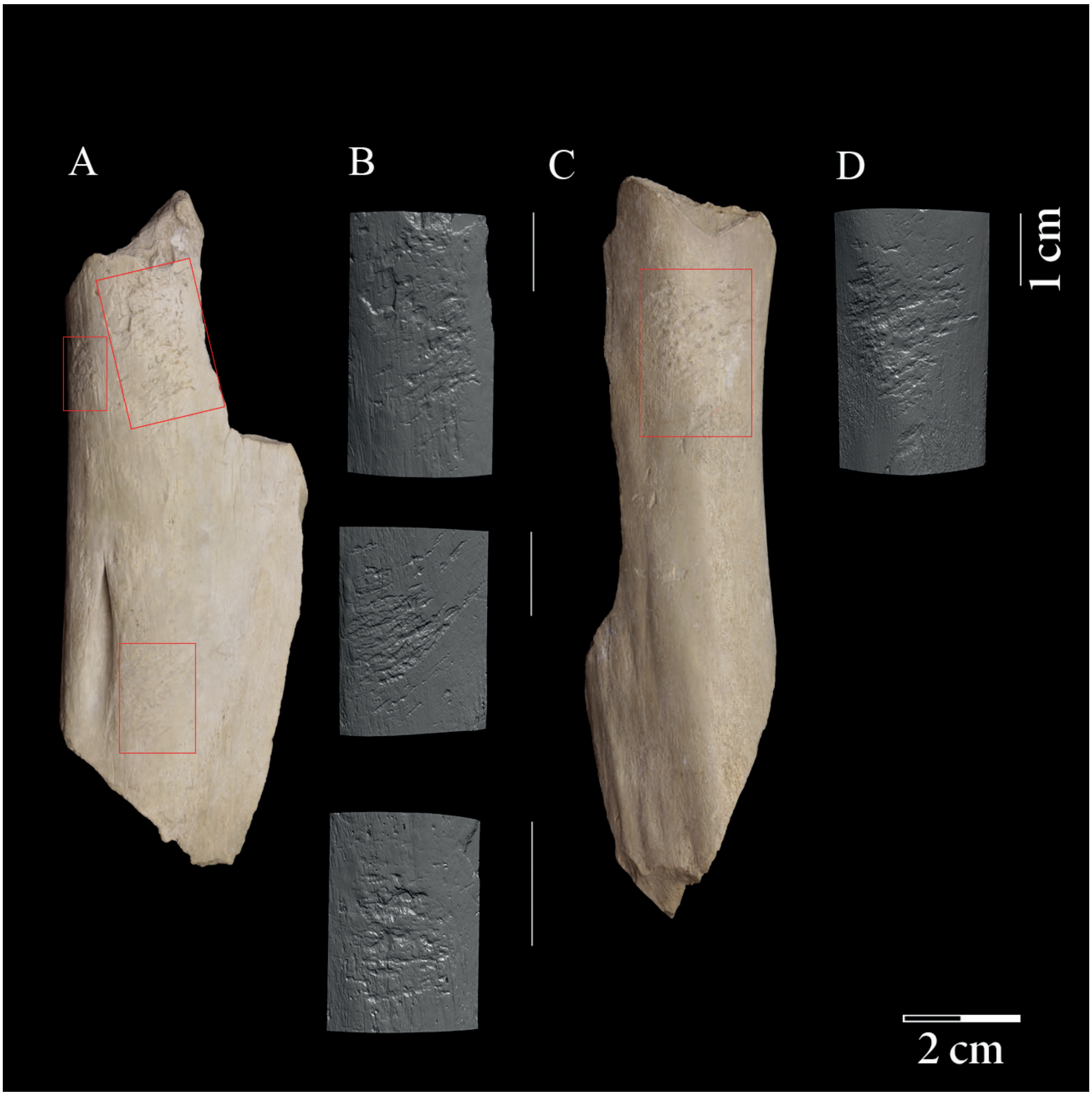

FIGURE 8. Bone retouchers associated with the Mousterian from Première Caverne du Bay Bonnet (Fonds-de-Forêt): (A) FDF_Ret02; (C) FDF_Ret01; (B, D) 3D scan of the use surfaces. Image G. Abrams (© RBINS).

a mass spectrometric precision of ±0.2‰ and ±0.3‰, respectively. C/N is the carbon to nitrogen atomic ratio. We corrected our AMS measurements on ultrafiltered collagen based on blanks subtracted at the combustion and graphitization stage, as well as the pre-treatment background subtraction (Wood et al., 2010). The subtraction for the HPLC derived background is based on the method detailed in Devièse et al. (2018).

3. Results

In this study, radiocarbon dates were obtained on ultrafiltered collagen for Mousterian industries from Trou du Diable, Trou Al'Wesse and Trou de l'Abîme and for potential Aurignacian from Scladina Cave (Tables 1-2; Fig. 10). Among those, the dates obtained on bone retouchers from Trou du Diable yielded the youngest ages for the Mousterian in Northwest Europe: 42,300–39,900 cal BP (TDD_RET32; OxA-34222; Fig. 7C) and 42,400–40,100 cal BP (TDD_RET62; OxA-34223; Fig. 7D). The potential Aurignacian from Scladina was dated between 41,700–39,300 cal BP (Sc2007-140-1; OxA-34044; Fig. 4A), therefore may be contemporaneous with the Late Mousterian. Echoing the work on Neanderthal bones from Belgium (Devièse et al., 2021), these dates produced using routine radiocarbon pre-treatments were possibly minimum ages due to the presence of unremoved contamination. It is indeed sometimes difficult to fully remove all contamination (e.g., from the sedimentary environment or from conservation materials) using the routine bulk collagen radiocarbon pre-treatments, particularly when the contaminants are potentially cross-linked to the collagen. Where possible,

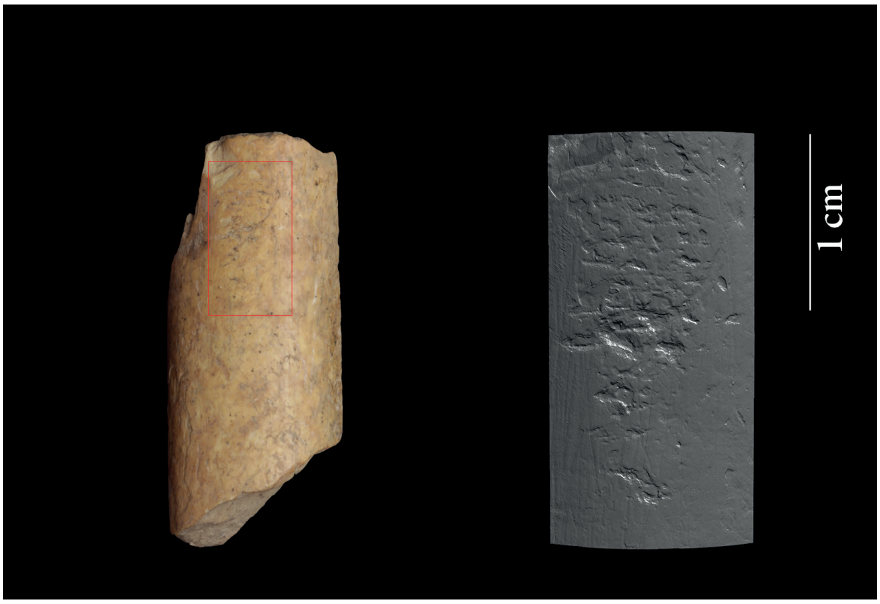

FIGURE 9. Bone retoucher (A; ENGIHOUL_Ret26) associated with the Mousterian from Gisement Paléolithique d'Engihoul. (B) 3D scan of the use surface. Image G. Abrams (©Les Chercheurs de la Wallonie).

the bone tools were cross dated, using the CSRA approach, isolating the amino acid hydroxyproline (HYP) from the bone collagen for AMS dating (see methods; Devièse et al., 2018a; Tripp et al., 2019).

The two retouchers from Trou du Diable, redated using the CSRA approach, provided significantly older dates than those obtained on collagen. Retouchers TDD_RET32 made of a reindeer bone and TDD_RET62 made of horse bone produced ages of 48,000–43,000 cal BP (OxA-X-2767-8) and >43,900 cal BP (OxA-X-2762-11), respectively (Tables 1, 3; Fig. 10). We dated two additional retouchers from Trou du Diable made of bovinae (TDD_RET40; Fig. 7A) and cervidae bones (TDD_RET64; Fig. 7B) using the same CSRA methodology. They produced ages of 49,800–42,300 cal BP (OxA-X-2762-26) and 47,100–42,800 cal BP (OxA-X-2767-16), respectively. A Chi-squared test run on the four uncalibrated HYP dates obtained on the retouchers from Trou du Diable yielded an error weighted mean of 42,028 ± 765 and a t value of 0.68. Since t is <7.81, the error weighted mean is not significant, and the four dates are therefore statistically identical. Similarly, we redated the two retouchers from Trou Al'Wesse using the compound specific approach. One retoucher made of cervidae bone (TAW545_RET01; Fig. 5A) produced an age of 52,600–43,300 cal BP (OxA-38323). The other retoucher (TAW545_RET03; Fig. 5B), made with of a horse bone produced an age >43,800 cal BP (OxA-38392). Dates obtained on collagen and hydroxyproline for this site are statistically identical by virtue of the sizes of the error bar on the ages. We also dated, using the CSRA approach only, two retouchers from Première Caverne du Bay Bonnet (Fonds-de-Forêt). The first, made of deer bone was dated to 48,700–42,900 cal BP (FDF_RET01; OxA-37773; Fig. 8A) and the second (FDF_RET02; Fig. 8B), made of horse bone, was dated twice, and produced ages of 50,300–42,900 cal BP (OxA-37796) and 48,000–42,700 cal BP (OxA-37797). Finally, we dated a bone retoucher from the Gisement Paléolithique d'Engihoul made of bear bone (Engihoul_RET26; Fig. 9). It produced an age >46,900 BP.

We applied the same compound specific approach to modified bones from Scladina Cave associated with an Upper Palaeolithic endscraper on a blade, based on their stratigraphic position, and two split-base points from Spy and Goyet caves, a tool type associated with the Early Aurignacian. The two modified bones from Scladina yielded ages of 41,100–37,200 cal BP (Sc83-312-1; OxA-X-2762-10) and 44,300–38,200 cal BP (Sc2007-140-1; OxA-X-2762-22). Because the bone retoucher (Sc2007-140-1; Fig. 4A) and the fractured fragment (Sc83-312-1; Fig. 4B) are presumed to be from the same bone, the dates could be combined (41,600–38,200 cal BP). The bone point from Goyet (IG-11735; SOM Fig. 2) gave an age of 40,200–37,500 cal BP (OxA-X-2767-15) and the one from Spy (Spy SP2-Spy1954; Fig. 3) an age of 42,100–40,300 cal BP (OxA-X-2767-14). This latest specimen was previously dated to 38,100–36,500 cal BP (GrA-32619), using a less robust pre-treatment approach (Semal et al., 2013a).

Radiocarbon dates obtained on bone collagen gave an estimated end for the Mousterian between 42,300-39,900 cal BP but the hydroxyproline dates obtained on the same objects gave significantly older ages (Fig. 10). We modelled, in a single-phase model, all the HYP dates

Site % Sample ID	Sample nature	P Code	% Yld	%C	δ13C (‰) (VPDB)	δ15N (‰) (AIR)	C/N	Conventional 14C Age	Lab number
Scladina cave									
Sc83-312-1	Percussion notches on a Horse proximal metapodial fragment	AF	4.8	44.0	-21.3	4.6	3.2	35,300 ± 650 B.P.	OxA-34045
Sc2007-140-1	Retoucher on a Horse distal metapodial fragment	AF	4.2	42.8	-21.1	4.9	3.2	35,350 ± 650 B.P.	OxA-34044
Trou du Diable									
TDD_RET32	Retoucher on bone shaft fragment of reindeer	AF	8.6	42.9	-18.8	3.2	3.2	36,250 ± 800 B.P.	OxA-34222
TDD_RET62	Retoucher on bone shaft fragment of horse	AF	8.7	39.7	-21.0	5.1	3.2	36,500 ± 800 B.P.	OxA-34223
Trou Al'Wesse									
TAW545_RET01	Retoucher on bone shaft fragment of cervid	AF	3.7	42.2	-19.9	5.5	3.2	45,700 ± 2,400 B.P.	OxA-34219
TAW545_RET03	Retoucher on bone shaft fragment of reindeer	AF	2.2	42.0	-19.1	2.6	3.2	41,300 ± 1,400 B.P.	OxA-34220
Trou de l'Abîme									
CTA H7 67	Retoucher one horse tooth	AF	1.6	43.0	-21.1	2.6	3.2	43,600 ± 1,900 B.P.	OxA-34120

TABLE 2. Radiocarbon dates obtained on bulk collagen at the ORAU. Conventional radiocarbon ages are expressed in years B.P. (Stuiver and Polach, 1977). PCode refers to pretreatment code; 'AF' is ultrafiltered collagen (Brock et al., 2010). %Yld is the percent yield of extracted collagen as a function of the starting weight of the bone analysed. %C is the carbon present in the combusted collagen sample. Stable isotope ratios δ13C and δ15N are expressed in per mil (‰) relative to vPDB and AIR, respectively, with a mass spectrometric precision of ± 0.2‰ (Coplen, 1994). C/N denotes the atomic ratio of carbon to nitrogen and is acceptable if it ranges between 2.9-3.5 in the case of collagen.

Site % Sample ID	Sample nature	P Code	% Yld	%C	δ13C (‰) (VPDB)	δ15N (‰) (AIR)	C/N	Conventional 14C Age	Lab number
Goyet Cave									
IG-11735	Split-base point made from reindeer antler	HYP	/	41.6	-24.0	3.1	5.1	34,000 ± 450 B.P.	OxA-X-2767-15
Scladina Cave									
Sc83-312-1	Percussion notches on a horse proximal metapodial fragment	HYP	/	39.3	-25.2	5.3	5.1	34,300 ± 800 B.P.	OxA-X-2762-10
Sc2007-140-1	Retoucher on a horse distal metapodial fragment	HYP	/	39.6	-29.3	3.3	5.1	30,150 ± 650 B.P.	OxA-X-2762-22
Sc2007-140-1	Retoucher on a horse distal metapodial fragment	HYP	/	40.1	-24.5	8.5	5.1	36,400 ± 1,600 B.P.	OxA-38390
Spy cave									
Spy SP2 - Spy 1954	Split-base point made from Reindeer antler	HYP	/	34.8	-21.3	6.5	5.1	36,200 ± 550 B.P.	OxA-X-2767-14
Trou du Diable									
TDD_RET40	Retoucher on bone shaft fragment of bison	HYP	/	42.2	-26.5	2.6	5.1	41,500 ± 1,800 B.P.	OxA-X-2762-26
TDD_RET64	Retoucher on bone shaft fragment of cervid	HYP	/	34.9	-26.0	3.1	5.1	41,700 ± 1,200 B.P.	OxA-X-2767-16
TDD_RET32	Retoucher on bone shaft fragment of reindeer	HYP	/	42.9	-22.6	5.0	5.1	42,300 ± 1,300 B.P.	OxA-X-2767-8
TDD_RET62	Retoucher on bone shaft fragment of horse	HYP	/	38.3	-26.0	6.3	5.1	44,100 ± 3,000B.P.	OxA-X-2762-11
Trou Al'Wesse									
TAW545_RET01	Retoucher on bone shaft fragment of cervid	HYP	/	44.3	-25.1	6.7	4.9	43,800 ± 2,100 B.P.	OxA-38323
TAW545_RET03	Retoucher on bone shaft fragment of reindeer	HYP	/	42.7	-22.0	6.9	5.1	45,100 ± 5,100 B.P.	OxA-38392
Gisement Paléolithique d'Engihoul									
ENGIHOUL_RET26	Bone retoucher on bone shaft fragment of cave bear	HYP	/	40.8	-25.9	4.9	5.0	> 46,900 B.P.	OxA-38393
Première Caverne du Bay Bonnet (Fonds-de-Forêt)									
FDF_RET01	Bone retoucher	HYP	/	54.4	-23.5	5.5	5.1	42,500 ± 1,400 B.P.	OxA-37773
FDF_RET02	Bone retoucher	HYP	/	39.5	-28.3	7.4	5.1	42,900 ± 1,600 B.P.	OxA-37796
FDF_RET02	Bone retoucher	HYP	/	45.1	-28.3	7.3	5.1	41,800 ± 1,400 B.P.	OxA-37797

TABLE 3. Radiocarbon dates obtained on the amino acid hydroxyproline at the ORAU. Conventional radiocarbon ages are expressed in years B.P. (Stuiver and Polach, 1977). PCode refers to pretreatment code 'HYP' that denotes the extraction of hydroxyproline from hydrolysed bone collagen (Devièse et al., 2018); %Yld are not reported because they were performed on collagen already extracted. %C is the carbon present in the combusted sample (hydroxyproline). Stable isotope ratios δ13C and δ15N are expressed in per mil (‰) relative to vPDB and AIR, respectively, with a mass spectrometric precision of ± 0.2‰ (Coplen, 1994). C/N denotes the atomic ratio of carbon to nitrogen and is acceptable if it ranges between 4.9-5.1 in the case of hydroxyproline. Specimen Sc2007-140-1 was dated twice because the first time it produced an abnormally young age (OxA-X-2762-22). We suspect that the sample was contaminated when or after it was graphitised. Only the second date (OxA-38390) is considered in the article.

obtained on bone retouchers from Trou du Diable, Trou Al'Wesse and Première Caverne du Bay Bonnet. The Bayesian modeling of the Mousterian bone retouchers points to an end boundary ranging between 45,900–42,900 cal BP (Fig. 11). This end boundary for the Mousterian in Belgium perfectly matches the chronological range of the latest Neanderthal occurrences in the same region (Spy 94a; 46,800–42,200 cal BP and Fonds-de-Forêt; 44,000–42,100 cal BP; Devièse et al., 2021; Fig. 3). These ages are slightly older than those modelled using AMS radiocarbon dating in (Higham et al., 2014; 41,030–39,260 cal BP).

The HYP dating of the modified bones excavated in Scladina Cave (41,600–38,200 cal BP; 95% probability) are coherent with those obtained on two split-base points from Spy Cave 42,100–40,300 cal BP (OxA-X-3767-14) and from Goyet 40,200–37,500 cal BP (OxA-X-3767-15). Therefore, the Upper Palaeolithic endscraper on blade from Scladina Unit T-RO can reasonably be associated with the Early Aurignacian although the artifact is not culturally diagnostic. The bone point from Spy Cave still constitutes the earliest clear Aurignacian evidence from Belgium, but the HYP age of 42,100–40,300 cal BP (at 95.4% probability) is much older than the age previously obtained on bone collagen 38,100–36,500 cal BP (GrA-32619; Pirson et al., 2012).

4. Discussion and conclusions

The coexistence of the different populations present on the European territory between 50,000 and 35,000 BP is a subject that is still strongly debated, especially since the addition of genetic data (Fu et al., 2015; Hajdinjak et al., 2021; Massilani et al., 2020; Prüfer et al., 2021; Slon et al., 2018). The (de)settlement dynamics of Northwest Europe is a complex phenomenon that we have approached through the chronology of archaeological and anthropological data. Although we are constrained by the relatively small amount of available remains, this dating project is unique in the quantity and diversity of remains studied as well as in the use of cross-dating to confirm our results.

These new data on Mousterian and Aurignacian industries change the narrative for the Middle to Upper Palaeolithic transition (MUPT) in the region. While the dates obtained on ultrafiltered collagen opened the possibility of a co-occurrence of the Mousterian and the Aurignacian cultures in Northwestern Europe, the HYP dates are pointing to a chronological hiatus between the end of the Mousterian (45,900–42,900 cal BP) and the earliest Aurignacian (42,100–40,300 cal BP). In other words, these results do not support the coexistence of different populations on the Belgian territory during the MUPT.

In addition, the new HYP results on the Mousterian bone industry, combined with the recently published HYP dates on the late Neanderthals, indicates the possible persistence of these Neanderthals beyond the latest dated Mousterian occurrences. This opens up the possibility of their association with the techno-complex LRJ. Although LRJ artefacts have been found from England to Poland, reliable chronological data are scarce (Flas, 2011b). In Belgium, this techno-complex has been found in Spy and Goyet but is totally absent from the caves of Engis and Fonds-de-Forêt, two of the sites that have yielded some of the most recent Neanderthals. Even if recent radiocarbon dates were obtained on bones from Nietoperzowa Cave (Krajcarz et al., 2018), they were produced on unmodified bones excavated mostly in the 1960s and cannot be considered as reliable referring to our methodology. So far, only the radiocarbon dating on collagen from the open-air site of Glaston (Cooper et al., 2012) can be qualified as the most reliable. These are around 44,000–42,000 cal BP. Based on this chronometric data, the Glaston LRJ would be contemporaneous with the Mousterian and thus with the last Neanderthals of North-western Europe, especially since we have no evidence of the presence of AMHs before 42,000–40,000 cal BP in this region. Moreover, we cannot exclude the possibility that the Glaston dates obtained on collagen could be minimum ages as dates obtained on collagen can be underestimates because of unremoved contamination as demonstrated on Neanderthals from Belgium (Devièse et al., 2021). So far, from England to Poland, there are no human remains that are firmly associated with LRJ

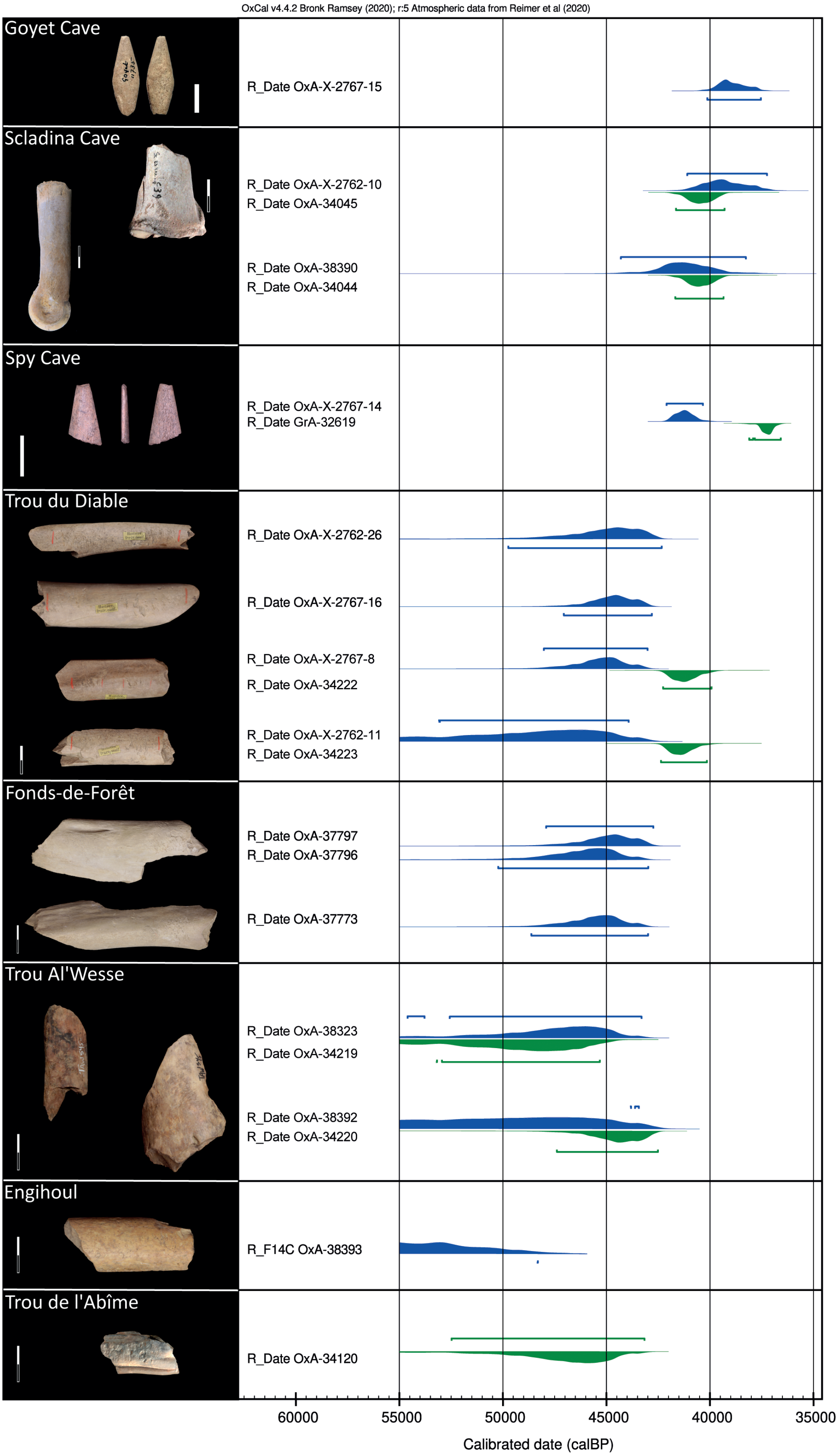

FIGURE 10. Calibrated age ranges of the bone points from Spy and Goyet caves and the retouchers from Scladina Cave, all related and/or associated to the Aurignacian and Mousterian bone tools from Trou du Diable, Première Caverne du Bay Bonnet (Fonds-de-Forêt), Trou Al'Wesse, Engihoul and Trou de l'Abîme. Dates obtained on collagen and hydroxyproline are reported in green and blue, respectively. The calibrated ages were obtained using OxCal 4.5 (Bronk Ramsey, 2009) and the IntCal20 calibration curve (Reimer et al., 2020).

industries, only a chronological convergence that seems to link this industry to those of the Late Middle Palaeolithic and the last Neanderthals. Based on current data, the LRJ could be considered as one of the several Late Middle Palaeolithic cultural facies. The model we propose, which highlights a hiatus between the Late Middle Palaeolithic and the Early Upper Palaeolithic, is therefore consistent with the current data available on LRJ industries. However, a link to the Initial Upper Palaeolithic cannot be firmly excluded (Demidenko and Škrdla, 2020). Several concomitant factors may explain the existence of a chronological hiatus observed in Belgium. In addition to the fact that we may not have discovered yet the very first Aurignacian productions or the very last Mousterian productions, North-western Europe is a remote region, at the edge of Neanderthal territories. This region is far from Central and Eastern Europe, the primary zones of AMHs influence around this period (Hublin, 2015; Hublin et al., 2020; Prüfer et al., 2021). In Northwest Europe, Neanderthals seemed to have evolved and gone extinct without any influence from modern humans. The climatic instability observed at that time in the Greenland records (Rasmussen et al., 2014) may have impacted the access to the faunal resources and contributed to the fragmentation of the territory and the declining demography that led to the isolation of last Neanderthal groups and, finally, to their extinction. This hypothesis is further reinforced by the absence of genetic introgression between North-western Neanderthals and AMHs (Hajdinjak et al., 2018).

It is also worth questioning the absence of a hiatus in other regions. This may sometimes be due, as we have observed in our study, to the dating methods employed. Radiocarbon dating is challenging, especially for periods close to the limit of the radiocarbon method and for objects that have been preserved in variable sedimentary conditions for thousands of years and received various conservation treatments which are not always well documented. In some cases, they are open to robust debate in terms of their reliability and accuracy. These challenges are rendered even more acute when we are exploring the dating of artefacts that are not exclusively linked with one hominin group, such as retouchers. Reliable dates are key if we are to understand more precisely the dynamics of the biocultural shift that took place over the course of the transition from the Middle to Upper Palaeolithic in Europe. Our results were generated using the CSRA approach which is chemically the most reliable means by which radiocarbon determinations on bone can be obtained. In several cases, as we have shown in this paper, dates of the same object using this approach and the more routine bulk collagen purification methods, are sometimes at odds with one another. Diagnosing which bulk dates from the literature are likely to be problematic is extremely difficult, owing to the absence of clear analytical parameters that signal when a date is inaccurate. Therefore, when undertaking large scale comparisons of radiocarbon dates obtained from the literature, we must necessarily proceed with a degree of caution. It would be appropriate, when possible, to confirm the chronological data obtained on bulk collagen with dates on hydroxyproline, as done, for example, at Kostenki (Dinnis et al., 2019), Vindija Cave (Devièse et al., 2017), and this study. New and more reliable models could then be built.

5. Supplementary information

5.1. Description of the archaeological contexts

5.1.1. Caves of Goyet

The caves of Goyet are in the municipality of Gesves (Province of Namur). The site consists of a vast cave network, in the Samson Valley, a tributary of the Meuse. The most famous cave is the Troisième Caverne that was first excavated by É. Dupont in between 1868 and 1870 (Dupont, 1872a). Since then, numerous other excavations have been conducted at the site by professional archaeologists and amateurs. In 1938, the Royal Belgian Institute of Natural Sciences conducted a second series of excavations led by the anthropologist F. Twiesselmann, in the backfill of Dupont and colleague excavations and in a small chamber known as the Salle du Mouton. During these excavations, a complete split-base point (Fig. 2) was excavated and dated here.

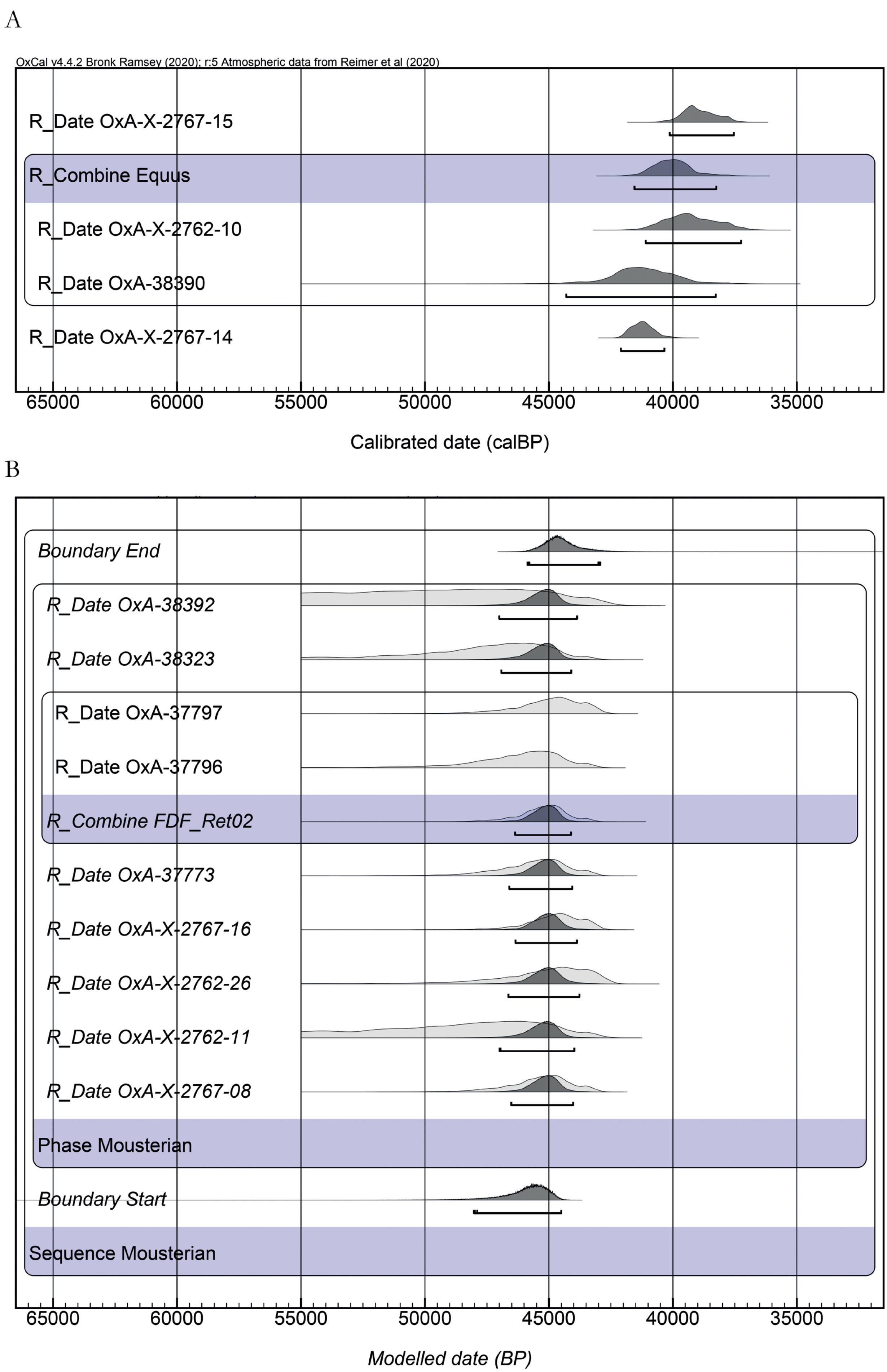

FIGURE 11. (A) Calibrated radiocarbon ages obtained on three Aurignacian industries; (B) Bayesian age model for the HYP dates of the Mousterian industries from Trou du Diable, Fonds-de-Forêt and Trou Al'Wesse. Engihoul is not included in the model as the age obtained is beyond the radiocarbon limit. The Mousterian data is modelled in a single-phase model. The code of the model is in the SI.

The caves of Goyet, together with Spy, is the most important Palaeolithic site in Belgium and of major importance on a European scale. It yielded numerous stone artefacts from the Middle and Upper Palaeolithic, as well as portable art and bone artefacts. From 2004 onwards, the reappraisal of the historical collections from the Troisième Caverne of Goyet has combined morphometric and taphonomic analyses together with direct radiocarbon dating as well as isotopic and genetic analyses (Hajdinjak et al., 2018; Posth et al., 2016; Rougier et al., 2016b). This study led to the identification of 100 Neanderthal remains, showing distinctive anthropogenic modifications (butchery activities and retouchers). Goyet is the first site where multiple Neanderthal bones were identified to have been used as retouchers, as well as the first site with unambiguous evidence of Neanderthal cannibalism in Northern Europe (Rougier et al., 2016b).

In addition to Neanderthal remains, the reappraisal of the Goyet collections led to the discovery of anatomically modern human remains that were directly radiocarbon dated to between 35,000 and 15,000 cal BP, making Goyet one of the richest palaeoanthropological sites in Europe, allowing the description of the major genetic transformations that took place in Europe throughout the Middle and Upper Palaeolithic in a single archaeological site (Fu et al., 2016; Posth et al., 2019).

The Troisième Caverne of Goyet delivered a large quantity of archaeological material related to the different cultures of the Middle Palaeolithic and the Upper Palaeolithic such as the Mousterian, the LRJ techno-cultural complex, the Aurignacian, the Gravettian, and the Magdalenian. It also delivered material from Neolithic and historical periods. However, their association with the human fossils is unclear given the lack of contextual data. The recent re-examination of the site's collections has demonstrated that the ossiferous levels described by É. Dupont represent a mix of materials from different periods (Rougier et al., 2016b).

5.1.2. Spy Cave

The Betche aux Rotches Cave, more commonly known as the Spy Cave, is in the village of Spy (Municipality of Jemeppe-sur-Sambre, Province of Namur). The cave opens south-west on the Orneau Valley, a tributary of the Sambre (Rougier and Semal, 2013).

The cave has been explored numerous times since 1879, when A. Rucquoy started the excavation of the inner part of the cave (Rucquoy, 1886-1887). After Rucquoy's excavations, numerous amateur archaeologists conducted investigations in the cave (Semal et al., 2013b).

The most important excavation campaigns were led by M. Lohest, a geologist from the University of Liège, and M. De Puydt, an enlightened amateur, in 1885–1886. They explored the terrace of the cave, which led to the discovery of the famous 'Men of Spy'. This was the first time that fossil human remains were found in a stratigraphic context in association with lithic material and extinct animal remains (De Puydt and Lohest, 1887b).

Between 1903 and 1909, A. de Loë and E. Rahir from the Royal Museums of Art and History of Brussels (RMAH), explored and drew a map of the cave, highlighting the different areas excavated by their precursors (de Loë and Rahir, 1911). They worked in the immediate surroundings of the De Puydt and Lohest excavations, digging on the terrace and the entrance of the cave, including the backfill of those previous excavations.

Between 1927 and 1933, excavations were conducted inside the Spy Cave by J. Hamal-Nandrin, C. Fraipont and S. Leclercq (Hamal-Nandrin et al., 1939), from the University of Liège.

In 1948 and from 1950 to 1954, F. Twiesselmann, Anthropologist, from the Royal Belgian Institute of Natural Sciences (RBINS), made a few test pits inside the cave (1950) and systematically excavated the slope in front of the cave (1952–1954), from the terrace to the Orneau River (Semal et al., 2013). This collection is the largest in terms of human, archaeological and paleontological remains. The fragment of bone point considered in this research was part of the collection excavated in

1954 and rediscovered among the reassessment of the faunal collection by Rougier and colleagues (Fig. 3; Flas et al., 2013).

The last official excavations attempting to locate in situ sediments were carried out by M. Dewez between 1979 and 1980 in the slope and inside the cave (Dewez, 1980, 1981).

Besides these officially reported excavations, numerous amateurs explored the site throughout the 19th and 20th centuries, excavating the backfill or in situ sediments in the cave, on the terrace or on the slope in front of the cave (Semal et al., 2013b). The latest significant discovery was an accidental incidental surface find in the early 2000's by a local, P. Pirson, who discovered a Neanderthal vertebra on the slope in front of the cave (Toussaint and Pirson, 2006).

5.1.3. Scladina Cave

The Scladina Cave is a large cave that opens on a zero-order tributary of the Meuse (Municipality of Andenne, Province of Namur). It was discovered in 1971 and has been under scientific excavation since 1978 (Bonjean et al., 2014b; Otte, 1992; Otte et al., 1998b).

A nearly complete juvenile mandible of a Neanderthal child was discovered inside the cave along with a fragment of right maxilla and isolated teeth (Toussaint et al., 2014). The remains of this ca. 8-year-old-child (Smith et al., 2014) were recovered in a secondary position in a gully. Combination of bone taphonomy, sedimentogenesis, paleoenvironmental reconstructions, and chronostratigraphic markers of the whole sequence points to an approximate age of either 87 or 80 ka, during a cold episode of the Weichselian Early Glacial (Pirson et al., 2014a).

Besides its anthropological interest, Scladina delivered interesting Middle Palaeolithic material related to two main Mousterian occupations (ca. 130,000 ago and ca. 43,000 cal BP), as well as several small Middle Palaeolithic lithic assemblages, and scattered Upper Palaeolithic pieces (Otte, 1998b; Pirson et al., 2018). It has also yielded a large quantity of bone tools made from Cave Bear remains (Abrams et al., 2014b) and the most north-western examples of black pigment importation and use by Neanderthals (Bonjean et al., 2015). In addition, Scladina yielded the most complete cave entrance sequence in Belgium (together with Walou Cave) making the site a reference for cave entrance contextual studies in North-western Europe (Pirson, 2014). The most recent chronostratigraphic interpretation of the sequence is based on climatostratigraphic, biostratigraphic, and mineralogical data, numerical dates, and comparison with the reference loess sequence from Central Belgium (Pirson, 2014).

While the site is primarily known for its Mousterian occupations and for the discovery of the Scladina Child, several lithic artefacts attest to the presence of an Upper Palaeolithic cultural component (Otte, 1998b). The sedimentary unit T-RO yielded a bone retoucher made of a horse metacarpal (Fig. 4A) associated with a fragment of a horse metacarpal bearing percussion notches (Fig. 4B). These bones, probably belonging to a single horse metacarpal, were the subject of the analysis presented here and are stratigraphically associated with an endscraper made on blade (Fig. 4C) that reinforced the presence of Upper Palaeolithic evidence.

5.1.4. Trou Al'Wesse

The Trou Al'Wesse is a cave located on the right bank of the Hoyoux, a tributary south of the Meuse River. This cave has been known to archaeologists since the 19th Century, a period during which it was notably excavated by É. Dupont (Dupont, 1873a) and by I. Braconier, in collaboration with M. Lohest and J. Fraipont (Fraipont and Braconier, 1887). This work revealed significant deposits, notably yielding Mousterian, Aurignacian and Neolithic elements (Otte, 1979; Ulrix-Closset, 1975).

From 1988, new excavations were carried out by the University of Liège and 'Les Chercheurs de la Wallonie', under the supervision of F. Collin. These excavations were mostly focused on the terrace in front of the cave, digging a long longitudinal trench (2 x 25m) which revealed the stratigraphic sequence, with Holocene deposits at the top overlaying a Late Pleistocene sequence down to the bedrock, including Aurignacian (Layer 15) and Mousterian (Layers 17a, b, c) occupations (Collin et al., 1996; Di Modica et al., 2005; Pirson and Collin, 2005).

From 2003, excavations continued under the direction of M. Otte and R. Miller to document more precisely the two phases of transition recorded at the site: the Mesolithic to Neolithic and the Middle to Upper Palaeolithic transitions. The first campaigns mainly focused on the Holocene part of the deposits, subdivided into several layers yielding Neolithic and Mesolithic material (Miller et al., 2012). From 2007 to 2016, it was mainly the Pleistocene deposits that were excavated, to record the environmental sequence corresponding to the end of the Upper Pleniglacial (e.g., Brace et al., 2012) and to study the levels yielding Aurignacian and Mousterian industries, before reaching the bedrock (Miller et al., 2015). Neanderthal DNA has been identified in Layer 17b sediments (Slon et al., 2017a). In 2018, the excavation of the deposits delivering the Mousterian assemblage was completed by a team led by D. Flas and N. Zwyns (Flas et al., 2019).

The retouchers studied and dated within the framework of this study come from Mousterian level 17 excavated by F. Collin in 1995–1996 (Fig. 5).

5.1.5. Trou de l'Abîme

The Trou de l'Abîme is in the Municipality of Couvin (Province of Namur). The site comprises a large cave as well as a vast terrace forming a rockshelter measuring 50 m long and 5 m deep, in the Eau Noire Valley, a tributary of the Viroin, in the Meuse Basin (Cattelain et al., 1986). The cave was excavated mainly in 1887 (Lohest and Braconnier, 1888) and 1902 (Maillieux, 1905). In 1905, a series of test pits were done on the terrace of the large rockshelter by A. de Loë (RMAH) leading to the discovery of leaf point artefacts (Lohest and Braconnier, 1888; Ulrix-Closset et al., 1988). Between 1984 and 1987, three test pits were done on the cave terrace (Cattelain et al., 1986; Pirson et al., 2009a). Only one of them delivered Palaeolithic stone artefacts and Pleistocene fauna as well as a Neanderthal tooth (Toussaint et al., 2010).

As the Trou de l'Abîme tooth is a worn lower right deciduous molar that was likely lost during the life of the child (Toussaint et al., 2010), it is almost impossible to directly date it without risking its complete destruction. Revaluation of the context of the tooth (Pirson et al., 2009a; Toussaint et al., 2010) led to an age based on two radiocarbon dates made on associated faunal material 46,820 ±3290 BP (Lv-1559) and 44,500 +1100/-800 BP (GrA-40444) consistent with the associated Mousterian lithic industry. However, the dates were not obtained from humanly modified bones. Recently, archaeozoological studies confirmed a single short-term occupation of the site (Abrams and Cattelain, 2014) and allowed to associate the human tooth to anthropogenically modified faunal remains that have been directly, including the bone retoucher that is part of this study (Fig. 6; 43,600 ± 1900 BP), and thus to propose this range of dates for the Neanderthal individual.

5.1.6. Trou du Diable

The Trou du Diable of Hastière also known as Caverne d'Hastière is located at the confluence of the valley of Féron, a small tributary of the Meuse and the Fond de Tahaux. The cave has different chambers and measures 25 m long, 2–8m wide and presents a significant slope (15 m). It opens North, on a wide terrace situated 60 m above the current thalweg of the river.

The site was discovered in 1871 and excavated firstly by É. Dupont, who collected the richest collection, which is stored in the Royal Belgian Institute of Natural Sciences in Brussels.

Dupont determined a stratigraphy composed by five different Ossiferous levels, where the two lowest seemed to contain only faunal remains (Hyena and Cave bear occupations; Rahir, 1925), overcome by those containing evidence for two Palaeolithic occupations spread throughout the different stratigraphic units (Ulrix-Closset, 1975). Based on a techno-typological approach, two cultural facies have been identified: the oldest related to the Late Mousterian (Di Modica, 2010) and the most recent to the Middle Aurignacian (Flas, 2008; Otte, 1979).

Several other excavation campaigns took place throughout the 20th Century and delivered similar but smaller assemblages with very similar stratigraphic observations (Di Modica, 2005). While the Aurignacian industry was made exclusively on flints (Di Modica, 2005), the Mousterian assemblage highlighted the use of more diverse lithic raw material as for example the quartz, quartzite, sandstone and phtanite (Di Modica, 2010).

Even though the flint was still the main raw material used by the Mousterian (66.5% of the industry), the scarcity of this important resource near the site must have had a direct impact on the economic strategy, involving an adaptation of the technological concepts: some retouched artefacts have been extremely reduced and smaller fragments were intensely sharpened (Di Modica, 2010). The use of bone retouchers seems particularly well suited for these types of activities. Therefore, it is not surprising that Dupont excavated one of the largest collections of bone retouchers for North-western Europe at Trou du Diable, which is constituted by no less than 295 retouchers made on animal remains, including those that are part of this study (Fig. 7; Abrams, 2018).

5.1.7. Fonds-de-Forêt Caves

The Bay Bonnet caves, also known as Fonds-de-Forêts caves, are in the Magne Valley (Municipality of Trooz, Province de Liège), a tributary of the Vesdre river. The two caves open on the left bank of this deep valley, just 1500 m upstream of the confluence and are separated from each other by a dozen meters.

They were first explored by P.-C. Schmerling between 1830 and 1831 and, from time to time, by collectors until 1895, when F. Tihon started his exploration of the site leading to the discovery of two human remains in the first cave (the most upstream; Tihon, 1898). He was also the first to provide stratigraphic observations for the two caves. After Tihon's excavations, numerous other scholars and collectors excavated the site until a short excavation campaign in 2003, which aimed to secure the cave (installation of grids).

Based on Tihon's observation, the Première Caverne is composed of 4 different layers. Layer 3 is the most important and contains most of the archaeological material and the two human specimens: a femur and an upper molar. This layer (named Layer F or 3rd Ossiferous Unit) was also recognized by A. Rutot, geologist, who excavated the site in 1907 (Rutot, 1909b). The archaeological material is associated with a Mousterian industry close to the one identified in La Quina (Ulrix-Closset, 1975). Based on its faunal remains, this layer has been attributed to the first half of the Marine Isotope Stage 3 (50,000–35,000 BP; Cordy, 1984). According to different scholars, this unit was covered by a layer that yielded artifacts from the Upper Palaeolithic such as the Aurignacian, the Upper Perigordian and the Ahrensbourgian (Dewez, 1987; Otte, 1979).

The material from the 3rd Ossiferous Unit should therefore be related to the Middle Palaeolithic and provided the bone retouchers dated in this research (Fig. 8). The Mousterian artefacts represent most of the archaeological material.

However, we prefer to remain cautious about the chronocultural attribution of the archaeological material and its association with stratigraphic units, especially because we are dealing with historic excavations and that the archaeological records were mixed within the different stratigraphic units and between the two different caves (Ulrix-Closset, 1975).

5.1.8. Gisement Paléolithique d'Engihoul

The Gisement Paléolithique d'Engihoul was in the Municipality of Engis (Province of Liège). The cave opened on the Engihoul stream, a small tributary of the Meuse. The site was discovered in 1931 and excavated by 'Les Chercheurs de la Wallonie' when a substantial part of the site seemed to have already been destroyed (Leruth, 1931). The terrace and the entrance of the cave were excavated until the destruction of the site by the progress of the quarry face (1938). The site was located at a height of about thirty meters above the bottom of the valley and consisted of a vast terrace about fifteen meters long and four to five meters wide when it was discovered (Leruth, 1931).

On the terrace, below the Aurignacian unit (Unit 8), three Mousterian units were excavated: the Levalloisien (Unit 6), the Moustérien typique (Unit 4) and the Moustérien supérieur (Unit 2) as well as several hearths (Vandebosch, 1933). Based on the stratigraphic description, there was only one Mousterian unit inside the cave which was an inner development of the Moustérien supérieur unit, which was identified on the terrace (Vandebosch, 1939a). The lithic material has been examined several times to clarify the chrono-cultural attribution. These studies were confronted with several difficulties such as an unrepresentative quantity of material available, the mixing of typological categories which does not consider stratigraphic origin (André, 1980-1982; Di Modica, 2006; Ulrix-Closset, 1975).

A recent reassessment of the collections has led to the identification of about 40 bone tools (Abrams, 2018) whose stratigraphic attribution and association with the different lithic industries are difficult to ascertain. The correlation between the material and their attribution can be made, in certain cases, by the presence of a marking on the material, which indicates the date of the discovery and thus the correspondence to the excavation notebooks which sometimes contain descriptions of the pieces found. The bone retoucher that was dated in this study is the only evidence of an ursid bone retoucher from this site (Fig. 9). Engihoul is thus the second Belgian Middle Palaeolithic site, along with Scladina Cave (Abrams et al., 2014b), to have provided evidence of the use of bear remains for functional purposes.

5.2. Code of the Bayesian model

```
Plot()
{
 Outlier_Model(«General»,T(5),U(0,4),»t»);
 Outlier_Model(«SSimple»,N(0,2),0,»s»);
Sequence(«Mousterian»)
{
 Boundary(«Start»);
 Phase(«Mousterian»)
 {
  R_Date(«OxA-X-2767-08», 42300, 1300)
  {
   Outlier(«General», 0.05);
  };
  R_Date(«OxA-X-2762-11», 44100, 3000)
  {
   Outlier(«General», 0.05);
  };
  R_Date(«OxA-X-2762-26», 41500, 1800)
  {
   Outlier(«General», 0.05);
  };
  R_Date(«OxA-X-2767-16», 41700, 1200)
  {
   Outlier(«General», 0.05);
  };
  R_Date(«OxA-37773», 42500, 1400)
  {
   Outlier(«General», 0.05);
  };
  R_Combine(«FDF_Ret02»)
  {
   Outlier(«General», 0.05);
   R_Date(«OxA-37796», 42900, 1600)
   {
    Outlier(«SSimple», 0.05);
   };
   R_Date(«OxA-37797», 41800, 1400)
   {
    Outlier(«SSimple», 0.05);
   };
  };
  R_Date(«OxA-38323», 43800, 2100)
  {
   Outlier(«General», 0.05);
  };
  R_Date(«OxA-38392», 45100, 5100)
  {
   Outlier(«General», 0.05);
  };
```

```
  };
  Boundary(«End of Mousterian»);
 };
};
```

5.3. Additional methods: 3D Scanning

The bone tools were digitized in 3D using the Keyence VR-5200. The Keyence VR-5200 is a 3D measurement system using structured light principles. It can stack different measurements in XYZ to capture tiles of a surface of 200 by 100 mm over 30 mm high. The image receiver is a monochrome 4Mpx CMOS. It uses 3 telecentric lenses, to capture the object from 3 different views, allowing to reconstruct an accurate 3D. The system offers 4 magnifications: x12, x25, x40 and x80. The bone tools were digitized using a x80 magnification. The measurement accuracy is ±2 µm on xy and ±4 µm on z and the resolution is 0.1 µm. The 3D model obtained from one view of the objects have been exported as .stl files and uploaded to Sketchfab for visualization.

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All the radiocarbon data generated at the ORAU are archived internally and are available upon request. ORAU data are also available on the laboratory's website, along with a link to the paper.

Author contributions

GA, TD, SP, TH and KDM designed the research. GA, CJ, IJ, PC, DB, AM and PS granted access to the collections, the archives and images. TD and TH performed the experimental work. GA, TD, SP, DF, PS, TH and KDM analyzed and interpreted the radiocarbon data. GA, TD, SP, IDG, DF, PS, TH and KDM analyzed the archaeological data. GA and TD wrote the manuscript with input from all authors.