Detecting stratigraphical issues using direct radiocarbon dating from small-mammal remains

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Running title: Radiocarbon dating small mammal remains

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Abstract

Frequently preserved in archaeological and paleontological sites, the tiny size of small-mammal remains favors percolations into underlying layers along stratigraphic sequences. This is one of the multiple post-depositional processes that may affect the integrity of the original deposits and therefore the subsequent scientific interpretations. Recent developments in sample preparation offer the possibility of detecting intrusive episodes through the absolute dating of minute amounts of bone (down to 10 mg), meaning that isolated elements (such as mandibles in this case) are sufficient to obtain reliable radiocarbon dates if collagen is moderately to well preserved. The radiocarbon dates obtained here for small-mammal bones (recovered from pre-Bølling to recent deposits) and their comparison with previous dates obtained from other sources (large-mammal bones, charcoal, botanical samples, etc.), with different protocols and instruments, illustrate the potential of small-mammal dating to reveal (and eventually contribute a solution to) stratigraphical issues in different archaeological contexts.

Keywords: archaeology; absolute dates; small mammals; stratigraphic sequences; intrusive episodes.

Introduction

Small-vertebrate remains are frequently preserved in archaeological and palaeontological deposits such as caves and rock shelters, sometimes in association with human remains and artifacts. Contrary to their larger counterparts, which are usually a product of human selection, the small-vertebrate accumulations mainly result from digestion or storing by their predators (i.e., birds of prey, small carnivores). Despite unavoidable filters due to specific predators (e.g., Mellet, 1974; Andrews, 1990), they reasonably well reflect local biocenoses. Presence/absence and relative abundances of small-mammal species can thus be used as proxies for biochronology (e.g., Cuenca-Bescós et al., 2010, 2015) and for the reconstruction of both past environments (e.g., López-García et al., 2014; Rofes et al. 2014, 2015; Royer, 2016) and biogeographical histories (e.g., Cucchi et al., 2014; Rofes et al., 2018).

Such interpretations are based on the working hypothesis of a strict association between small vertebrates and archaeological remains, implying a good archaeological coherence of the analyzed assemblages. However, this is not always the case. Multiple kinds of post-depositional processes can affect the stratigraphy of a given site by altering the integrity of the deposits (e.g., Wood and Lee Johnson, 1978; Texier, 2000) and therefore the subsequent scientific interpretations. For instance, caves and rock shelters are propitious places for small and medium size animals to nest or build burrows, which may significantly modify and pollute faunal assemblages (Dalland and Carter, 1998; Mallye, 2011; Pelletier et al., 2016).

Absolute dates are crucial for the reconstruction of past environments: they constitute diachronic anchors for the signals tracing the evolution of climate and habitats in a given location. They are also essential for biochronology and biogeography, to explore the accuracy of chronological biomarkers, in the first case, and to test the reliability of previous hypothesis (e.g., from phylogeography), in the second. Finally, they allow the evaluation of faunal assemblages regarding the contemporaneity of archaeological remains and species (e.g., Costamagno et al., 2016; Pelletier et al., 2017; Royer et al., 2018).

Accelerator mass spectrometers (AMS) have revolutionized the field of archaeology since their inception, allowing absolute dates to be obtained for the last 50 ky (Reimer et al., 2013). However, routine AMS dating still requires 60-200 mg of bone, depending on preservation state, which far exceeds the mass of isolated elements of the skeleton of many small mammals, such as rodents, shrews and bats. Here, we present 15 radiocarbon dates obtained from minute amounts of bone (less than 25 mg) of small mammals, using a Mini Carbon

Dating System (MICADAS) and following a recently developed optimized protocol (Cersoy et al., 2017a, 2017b). The samples come from four archaeological sites with different chronologies, chrono-culturally spanning from the upper Magdalenian to recent times. We compare these new dates with previous ones obtained from different materials (e.g., large- and small mammal bones, charcoal, plants) in different laboratories, using other protocols, to check for agreements. Departing from this comparison, our purpose is to evaluate how and to what extent this new procedure can detect stratigraphical issues in archaeological sequences and contribute to their solution.

Materials and Methods

Archaeological sites

The following sites are all located in rock shelters at different altitudes, with the exception of Beg ar Loued, which is an open-air household settlement next to the coast. The location of the sites is shown in Figure 1.

The site of **Peyrazet** (Creysse, Lot) has five stratigraphic levels, chronologically spanning from the pre-Bølling period (end of Heinrich Stadial 1) to the Preboreal. The site contains archaeological material from the recent Laborian (=Final Paleolithic; a lithic techno-complex occurring during the transition between the Younger-Dryas and the Preboreal in southwest France [Langlais et al., 2015]), the Azilian (during the Allerød chronozone), and the Upper Magdalenian (during the Bølling) (Langlais and Laroulandie, 2016). Eight radiocarbon dates from reindeer, roe deer and water vole bones confirmed this chronology (Langlais et al., 2015; Costamagno et al., 2016; Royer, 2016).

The settlement of **Beg ar Loued** (Molène Island, Brittany) was built during the Early Bronze Age. The radiocarbon dated shrew mandible here comes from the infill of a dry-stone oval building, which, according to several radiocarbon dates obtained from other sources (charcoal and large-mammal bones for the most part), was likely occupied from 2200 to 1800 years cal BC (Pailler et al., 2014, 2019).

The rock shelter of **Lano** (Castagnicia, Haute-Corse, Corsica) was used for funerary purposes, as revealed by the presence of three wooden coffins found inside. The coffins contained human remains dating to the Bronze Age, based on radiocarbon dating of the

human bones and wood from a coffin (Leandri et al., 2016). In 2016, a test pit was conducted at roughly 2 m from the entrance of the cave. The stratigraphic section was divided into stratigraphic units (US), which, from bottom to top of the sequence, were named US 12 to US 5. The layers D1-D5, from a previous test pit made in 2015, were correlated with US 5 and 6 of this new stratigraphy, being D1 the uppermost and D5 the lowest.

The **Grande Rivoire** site (Sassenage, Isère, Auvergne-Rhône-Alpes) has been divided in several excavation sectors. The small mammal remains dated in this study come from the NR16-21 and SU16-22 sectors. They comprise five different chrono-cultural units, listed here from earliest to latest: Second Mesolithic, transition from Second Mesolithic to Early Neolithic, Early Neolithic, Middle Neolithic 1 and Middle Neolithic 2. These units were subdivided in turn into subunits ("décapages") and several radiocarbon dates from charcoal, large-mammal bones, seeds and hazelnut were obtained for them, all supporting the chronology (Rofes, 2018).

Samples

Fifteen mandibles of shrews (Soricidae, Mammalia) from the above-mentioned locations have been radiocarbon dated (Table 1). The mandibles belong to the species *Crocidura suaveolens*, *C. russula*, *C. leucodon*, *Neomys anomalus* and *Sorex* gr. *araneus-coronatus*, weighing between 10.5 and 25.0 mg as displayed in Table 2. They were part of natural accumulations of small vertebrates, mainly contributed by birds of prey (i.e., rejection pellets). The taxonomical identifications were performed by one of us (JR), based on diagnostic morphological characters.

Sample selection: optimized sample yield and ATR-FTIR estimation of collagen content

Prior to radiocarbon dating, we estimated the collagen content of the samples using attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), following the procedure described in Lebon et al. (2016). Due to their small size, only three samples could be examined (JR1, JR 16 and JR 24, Table 1). Briefly, less than one mg of crushed bone was placed on the diamond ATR accessory (Golden Gate Single Reflection Diamond

ATR accessory, Specac, France) and analysed with a Vertex 70 FT-IR spectrometer (Bruker Optics, France).

ATR spectra (Figure 2) were obtained by the accumulation of 128 scans in the wavenumber range 4000–400 cm^{-1} with a spectral resolution of 2 cm^{-1}. During acquisition, ν_3PO_4 peaks were normalized to an absorbance of 0.5. Data treatment (linear baseline correction and amide I /phosphate $\nu_3(PO_4)$ bands measurement) was performed using OPUS software (Bruker Optics, France). Collagen contents were estimated from the amide I/PO_4 ratio using equations reported in Lebon et al. (2016), taking standard deviations into account. Note that this method is not reliable for quantification below the 4% yield threshold, and collagen detection is impossible below 3%.

The amount of bone sample needed to perform the radiocarbon date can be predicted using a chart (Figure 3). In the X-axis, the collagen yield in % is reported. Using the 0.2 mgC curve, which is the threshold for the commercially available AGE3 graphitization unit, the minimum amount of bone required for a radiocarbon date can be read on the Y-axis. Two more curves are reported, corresponding to 0.5 mgC and 1 mgC: they show the limit for radiocarbon dating on (more widespread) conventional AMS. This limit gives the amount of bone needed at worst to obtain a radiocarbon date: 18 mg for MICADAS dating (after graphitization) and 40 mg for conventional AMS dating. On the other hand, maximum collagen content in modern fresh bones is indicated (20-25 %) (Schoeninger et al., 1989).

Collagen content for the three samples varies between 5.2 and 8.0 % indicating that a minimum amount of 6.3 to 9.6 mg of bone is required, if all the bone collagen content is extractible (meaning that the collagen content is equal to the collagen extraction yield), to obtain the minimum 0.2 mg of carbon for radiocarbon dating analysis on ECHoMICADAS (dotted curve). As mandibles weighed between 10.5 and 25 mg (as stated in Table 2), we applied the extraction protocol to all the samples.

Collagen extraction and radiocarbon dating

Collagen extraction and radiocarbon dating were performed following Cersoy et al. (2017a, 2017b). Briefly, mandibles were immersed in 0.2 M hydrochloric acid at 4 °C for 2 days to perform demineralization. The remaining decalcified collagen was rinsed with Milli-Q water and purified in 0.1 M NaOH at 4 °C for 2 days to eliminate soil contaminants. Finally,

collagen was rinsed with 0.2 M HCl and then Milli-Q water before gelatinization in 0.06 M hydrochloric acid at 90 °C for one hour, and purification by glass filtering. For this last step, specific glass vacuum filtration devices, with 1.6 microns maximum pore size, where designed and manufactured by Ellipse (France). Freeze-dried collagen was transferred to tin capsules after resuspension in ultrapure water and combustion was performed in an Elemental Analyzer (EA). All samples, except JR 15, were graphitized on the commercial compact graphitization system AGE 3 (Ion plus, Switzerland) (Wacker et al., 2010a). Due to the low amount of collagen extracted from JR 15 bone sample, a gas interface system (GIS) was used (Wacker et al., 2013). Following combustion in the EA, sample CO_2 was adsorbed on a zeolite trap before being released and expanded to the syringe of the GIS (Ruff et al., 2010; Wacker et al., 2013). For all samples, dating was performed on the compact AMS ECHoMICADAS at Gif-sur-Yvette (France) (Wacker et al., 2010b).

Results

The radiocarbon dates of the samples are reported in Table 2. Collagen yields range between 3.1 and 15.4 %, above the 1% threshold for datable samples, confirming the moderately to good preservation of the collagen in the bone samples. We were thus able to extract enough collagen for radiocarbon dating (corresponding to 0.188 to 0.862 mgC). C/N ratios range between 3.2 and 3.6, within the 2.9-3.6 limits suitable for radiocarbon dating (DeNiro, 1985; Van Klinken, 1999).

Peyrazet

Comparing the dates in this study with those previously obtained for Level 4 and Level 2 (Table 3), we observe that, with regard to Level 4, there is an agreement between our dates (12940±70 14C a BP, 12960±70 14C a BP) and the one previously performed on a selection of water vole (*Arvicola* sp.) bones dated altogether (12960±70 14C a BP; Royer, 2016) in another laboratory (Lyon) with conventional extraction and classical AMS dating. The rest of the dates available for this unit (Ly-6437 to Ly-13447 in Table 3) are also consistent with the new ones and with the chrono-cultural attribution (Upper Magdalenian).

However, there is a significant difference in Level 2 between our dates (1585±30 14C a BP, 1475±30 14C a BP) and the one previously performed on a metapodial of red deer (*Cervus*

elaphus) considered to be *in situ* (9780±45 14C a BP; Langlais et al., 2015). Another date newly obtained for the site (11790±230 14 C a BP) is also in agreement with a previous one obtained for a roe deer (*Capreolus capreolus*) metacarpal (11810±50 14C a BP), both remains from the same stratigraphical interface between levels 3 and 4.

Beg ar Loued

A mandible of *Neomys anomalus* gave a date of 3650±30 14C a BP (ECHo-1258). This date is somewhat older than a previous one, performed on a *Microtus agrestis* mandible (3177±35 14C a BP, UB-6925) found in the same scree layer inside the house (Table 3) (Pailler et al., 2019).

Lano

The comparison of our dates (Table 3) for layers D4 (post-bomb) and D1 (515±20 14C a BP) with those previously obtained from the Poznan Radiocarbon Laboratory for layers D5 (post-bomb) and D3 (1155±30 14C a BP), the latter two from selections of small-vertebrate bones with a different technique, allows us to state that: 1) layers D1 and D3 are in good stratigraphical order, D1 dating to the 15th century and D3 to the 8th-10th centuries; 2) D4 and D5 are modern; 3) these data in turn allow to infer that the sequence D5-D1 presents a "stratigraphic inversion", where layers D4 and D5 are more recent than D3-D1; and 4) considering that the US 6 and 5 nearly coincide with layers D5-D1 (see Lano section on Materials and Methods), these units may also present a stratigraphic inversion.

Grande Rivoire

Comparing our seven dates to the *corpus* previously obtained from other materials (i.e., large-mammal bones, charcoal, plants), by different laboratories (Lyon-Saclay and Beta Analytics), we observe that they all agree with the general chronostratigraphic framework. Some of our dates (5070±40 14C a BP [d88], 5760±40 14C a BP [d111], 6510±40 14C a BP[d142]) are almost identical to those already obtained on the same layers (see Table 4), whereas others (7130±40 14C a BP [d143], 7600±50 14C a BP [d147], 6750±50 14C a BP [d28]) are a few centuries older without being in contradiction with the general framework. Finally, it should be noticed that the date obtained for the layer d22 of the sector SU16-22 (6410±45 14C a

BP), attributed to the early Neolithic by the excavators, is also coherent with that of the layer d26 (6510±40 14C a BP), which is directly underlying.

It should be noted that three of the oldest dates appear to have a higher C/N ratio, above 3.5. Nonetheless, hardly this can explain the discrepancy observed for two reasons: (1) usually, contamination makes the sample appear younger (and not older) because contaminants come from the soil organic matter whose 14C activity is younger than the bone remains (as recently demonstrated in Zazzo et al., 2019) and (2) other samples have also high C/N ratio but they are in good agreement with the paired date, like, for instance, JR 16 (from Peyrazet, described above), which is older and therefore more prone to contamination. Hence, we think the discrepancy observed is rather due to a stratigraphic issue at Grande Rivoire rather than to contamination.

Discussion

In Peyrazet, the perfect agreement between the new dates and those previously obtained in another laboratory for Level 4 (Upper Magdalenian) confirms the reliability of the new protocol. At the same time, these direct dates attest the oldest postglacial presence of *N. anomalus* in western Europe (see Table 3). The two dates from the interface between Levels 4 and 3 (Upper Magdalenian/Azilian), confirm one another despite the large uncertainty associated with our measurement (±230 14C a) for the mandible of *C.* cf. *suaveolens* (due to the low amount of extractible collagen). To our knowledge, this is the oldest direct postglacial evidence (i.e., 14175-13129 cal a BP) for this taxon in western Europe up to now, giving an age well within the Bølling-Allerød warm pulse (c. 14700 to c. 12700 a BP). Notably, it confirms a recently proposed scenario for the postglacial recolonization of this species from the Iberian Peninsula (Rofes et al., 2018). There is a notorious lack of absolute dates for small mammal taxa to support postglacial recolonization models inferred by other means (e.g., molecular phylogeography), thus those recently obtained for the *N. anomalus* and *C.* cf. *suaveolens* of Peyrazet will certainly be of great biogeographical and biochronological value.

The difference between our dates and those previously obtained regarding Level 2, can be explained by the presence of bioturbation in upper deposits. The archaeological materials seem relatively homogeneous from a cultural standpoint, although protohistoric ceramics have been found in these upper levels (Langlais et al., 2015). The integrity of the small vertebrate assemblages was questioned due to the association of black rat (*Rattus rattus*) and European hamster (*Cricetus cricetus*) remains (Langlais et al., 2015). If the first species is clearly not attested in France before the Antiquity (Pascal et al., 2006), the second one suggests that these materials could be Younger-Dryas in origin, as it has been recently demonstrated for the hamster remains in Combe Cullier (Royer et al., 2018). The recent radiocarbon dates obtained from the two shrew remains (c. 5^{th} – 7^{th} centuries AD) thus strongly suggest the presence of more recent intrusive remains in this level, presumably brought from upper layers by burrowing activities. Further inferences made from this level should then be taken with caution.

Figure 4 displays the stratigraphy of Peyrazet (4A) and synthetizes all our findings (4B), including agreements and disagreements among dates.

The discrepancy of dates in Beg ar Loued may be due to the different periods in which the remains were naturally deposited inside the structure. The *Neomys* mandible could date from the foundation of the building during the Early Bronze Age, whereas the *Microtus* mandible could date from the time of abandonment of the household during the Middle Bronze Age. There are several potential reasons for their coexistence in the same scree level, including the activity of burrowing animals. Another straightforward explanation is that the shrew mandible was deposited over an elevated stone-wall which posteriorly crumbled.

In Lano, the presence of *R. rattus* in the lowermost levels of the sequence (US 12), indicates that the accumulation postdates the 4th-2nd centuries BC, a period during which the black rat was introduced into Corsica (Pascal et al., 2006). The absence of endemic taxa, such as *Prolagus*, *Asoriculus*, *Rhagamys* or *Tyrrenicola*, also indicates that this deposit is contemporary or postdates the extinction of the native fauna of the island between the 2nd and the 13th centuries AD. The stratigraphic sequence studied is thus clearly posterior to the deposit of the wooden coffins, which date from the first millennium BC (Leandri et al., 2016). It is too early to propose an explanation regarding the "stratigraphical inversion" detected. Works on the site continues and excavators will be reaching the end of the cavity soon. That will allow identification of the initial area of the deposit and perhaps the reason for the inversion, a phenomenon which is not surprising given the complexity of sedimentary processes in caves.

In Grande Rivoire, the seven ECHo dates are consistent with the general chrono-stratigraphy of the deposit (see Table 4 where they are displayed in stratigraphic order), even if there are three (ECHo-1247, ECHo-1696 and ECHO-1702) which are older (~3 centuries) than the measurements made on hazelnuts (Lyon-13971 and Lyon-13972) or bones (Beta 28246 and Beta-255118) from the same levels. For the two results dating back to the Mesolithic/ Neolithic transition (ECHo-1247 and ECHo-1702), the lag of a few centuries could be due to the slow formation of the levels in question, well evidenced by the sedimentological analyzes (Nicod et al., 2012). However, the good general coherence between the new dates and those previously obtained with other techniques, accredits also in this case the reliability of the new protocols. Moreover, these new dates show that the natural accumulations of small vertebrates in the different units do not result from modern contamination or post-sedimentary rearrangements. They instead come from *in situ* accumulations contemporary to the formation of the deposits. This validates any further stratigraphic, biogeographical and/or palaeoenvironmental interpretation made from the small-mammal associations of this site. It

should be mentioned that the dates obtained from charred hazelnuts (layers d143 and d147) are clearly more recent than the new dates from the same layers. This might be explained either by contamination or intrusions from upper units.

Conclusions

The radiocarbon dates obtained here for small-mammal bones and their comparison with previous dates obtained from other sources (e.g., large-mammal and other small-vertebrate bones, charcoal, botanical samples, etc.), with different protocols and instruments, show the potential of small-vertebrate dating to reveal (and eventually contribute a solution to) stratigraphical issues in different archaeological contexts, from pre-Bølling to recent times.

Our results show that chronological, environmental and/or biogeographical inferences based on the small-mammal assemblages from Levels 3 and 4 of Peyrazet, and the entire stratigraphical sequence of Grande Rivoire, are trustworthy. We cannot state the same for Level 2 of Peyrazet, the infill of the structure of Beg ar Loued, or the stratigraphy of Lano. Caution is advisable in the latter cases.

The tiny size of small-mammal remains favors percolations into underlying layers along stratigraphic sequences: this is a well-known phenomenon in archaeological deposits. The new technique offers a straightforward possibility of detecting intrusive episodes through the absolute dating of minute amounts of bone (down to 10 mg in this study), meaning that isolated elements (such as shrew mandibles in this case) are enough to obtain reliable radiocarbon dates if collagen is moderately to well preserved. Besides, this technique is far less destructive than previous ones, which require a minimum of 60 mg of bone to obtain results, meaning collections of bones of small vertebrates with the possible admixture of non-contemporaneous materials. Moreover, for bones of sufficient size, an ATR-FTIR prescreening can estimate quickly, with just 1 mg of bone, whether a sample contains sufficient collagen to be successfully radiocarbon dated or not, well before any further extraction or intense laboratory pretreatment.

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Lab code	Site	Taxa	Archeological context	Collagen content estimation (wt%)
JR 27	Lano	*Crocidura suaveolens*	Lano 2015 sondage D4	
JR 26	Lano	*Crocidura suaveolens*	Lano 2015 sondage D1	
JR 1	Beg ar Loued	*Neomys anomalus*	BAL04 SDII H3 p.2	7.1
JR 20	Grande Rivoire	*Sorex* gr. *araneus-coronatus*	GR05.O17a.d88.LJB (Sect. NR16-21)	
JR 21	Grande Rivoire	*Sorex* gr. *araneus-coronatus*	GR07.P16a.d111.LBCG (Sect. NR16-21)	
JR 18	Grande Rivoire	*Sorex* gr. *araneus-coronatus*	GR08.S20d.d22.LGF (Sect. SU16-22)	
JR 23	Grande Rivoire	*Sorex* gr. *araneus-coronatus*	GR13.Q17c.d142.LBl(B) (Sect. NR16-21)	
JR 30	Grande Rivoire	*Crocidura leucodon*	GR08.T17b.d28.LGM(F) (Sect. SU16-22)	
JR 24	Grande Rivoire	*Sorex* gr. *araneus-coronatus*	GR14.R16b.d143.LGM(C)/CX (Sect. NR16-21)	8.0
JR 31	Grande Rivoire	*Crocidura suaveolens*	GR14.P17d.d147.GR/LJBl (Sect. NR 16-21)	
JR 13	Peyrazet	*Crocidura russula*	PRZ10 M5C d.19 Level 2	
JR 14	Peyrazet	*Crocidura russula*	PRZ10 M5C d.20 Level 2	
JR 15	Peyrazet	*Crocidura* cf. *suaveolens*	PRZ09 L6C d.8 Interface Level 3/4	
JR 17	Peyrazet	*Neomys anomalus*	PRZ13 L6C d.32 Level 4	
JR 16	Peyrazet	*Neomys anomalus*	PRZ12 L6C d.28 Level 4	5.2

Table 1. Description of the samples, all of them mandibles of shrews. Collagen preservation in the sample was estimated using ATR-FTIR spectroscopy (see details in the 3rd section of Materials and Methods).

		Pretreatment			**Combustion**				**14C measurement**		
Lab code	**Site**	**Sample size** (mg)	**Collagen amount** (mg)	**Yield** (%)	**%C**	**%N**	**C/N**	**Carbon mass (**µgC)	**ECHO n°**	**14C age** (BP)	**Error**
JR 27	Lano	17.8	2.7	15.2	33.1	12.0	3.2	772	1252	-440	25
JR 26	Lano	25.0	3.6	14.4	33.6	12.0	3.3	862	1251	515	20
JR 13	Peyrazet	11.7	1.8	15.4	28.7	10.0	3.4	361	1259	1585	30
JR 14	Peyrazet	14.2	1.8	12.7	29.4	10.4	3.3	486	1245	1475	30
JR 1	Beg ar Loued	16.2	1.5	9.3	33.7	11.9	3.3	391	1258	3650	30
JR 20	Grande Rivoire	12.0	1.3	10.8	29.1	10.0	3.4	309	1262	5070	40
JR 21	Grande Rivoire	15.0	1.7	11.3	29.1	10.3	3.3	433	1256	5760	40
JR 18	Grande Rivoire	11.5	1.0	8.7	26.6	8.5	3.6*	188	1261	6410	45
JR 23	Grande Rivoire	10.8	1.4	13.0	31.3	10.6	3.4	232	1260	6510	40
JR 30	Grande Rivoire	10.5	0.9	8.6	18.4	6.1	3.5*	390	1702	6750	50
JR 24	Grande Rivoire	15.9	1.1	6.9	22.8	7.9	3.4	357	1247	7130	40
JR 31	Grande Rivoire	12.9	0.7	5.4	19.8	6.5	3.6*	323	1696	7600	50
JR 15	Peyrazet	12.3	0.4	3.3	15.5	5.4	3.3	174	1633	11790	230**
JR 17	Peyrazet	15.6	1.2	7.7	24.3	8.4	3.4	231	1253	12 940	70
JR 16	Peyrazet	22.8	0.7	3.1	30.7	10.2	3.5*	266	1254	12 960	70

Table 2. Radiocarbon dating of the shrew samples. Yield is estimated as the ratio (in %) of the total amount of collagen recovered from the amount of initial bone used for extraction. The carbon mass corresponds to the amount of carbon detected following combustion in the Elemental Analyzer and used to produce the graphite target. ECHo n° corresponds to the target numbers. *Four dates (three from Grande Rivoire and one from Peyrazet) appear to have a C/N ratio equal to or higher than 3.5. **Due to the low amount of collagen for this sample (JR 15), 14C content was measured via a gas interface system (GIS) which explains the higher value of the error on the 14C age.

Site	Cultural attribution	Context	Lab code	Element	14C date (BP)	2-Sigma cal BP	2-Sigma cal AD-BC
Peyrazet	Early Middle Age	Unit 2	ECHo-1259 (JR 13)	Shrew mandible	1585±30	1407-1542	AD 408-543
		Unit 2	ECHo-1245 (JR 14)	Shrew mandible	1475±30	1307-1407	AD 543-643
	Recent Laborian	Unit 2	Ly-7828 (SacA-22775)	Red deer bone	9780±45	11257-11141	BC 9308-9192
	Upper Magdalenian/Azilian	Interface between units 3 and 4	ECHo-1634 (JR 15)	Shrew mandible	11790±230	14175-13129	BC 12226-11180
		Interface between units 3 and 4	Ly-7826 (SacA-22773)	Roe deer bone	11810±50	13756-13541	BC 11807-11592
	Upper Magdalenian	Unit 4	Ly-6437 (SacA-17857)	Reindeer bone	12580±80	15208-14402	BC 13259-12453
		Unit 4	Ly-6436 (SacA-17856)	Reindeer bone	12720±80	15405-14776	BC 13456-12827
		Unit 4	Ly-13448 (Sac-A47545)	Chamois bone	12810±60	15536-15091	BC 13587-13142
		Unit 4	Ly-13447 (SacA 47544)	Reindeer bone	12840±60	15574-15126	BC 13625-13177
		Unit 4	ECHo-1253 (JR 17)	Shrew mandible	12940±70	15731-15228	BC 13782-13279
		Unit 4	Ly-11974 (Sac-A40416)	Water vole bones	12960±70	15752-15249	BC 13803-13300
		Unit 4	ECHo-1254 (JR 16)	Shrew mandible	12960±70	15752-15249	BC 13803-13300
	pre-Bølling	Unit 5	Ly-11975 (SacA40417)	Water vole bones	13540±80	16599-16052	BC 14650-14103
Beg ar Loued	Middle Bronze Age	SDII G/H/I3 US2004	UB-6925	Vole mandible	3177±35	3467-3342	BC 1518-1393
	Early Bronze Age	SDII H3 p.2	ECHo-1258 (JR 1)	Shrew mandible	3650±30	4014-3888	BC 2065-1939
Lano	Modern	D4	ECHo-1252 (JR 27)	Shrew mandible	-440±25	-4-0	AD 1956-1957
		D5	Poz-81616	Small vertebrate bones	-100±26	-4-0	AD 1955-1956
	Late Middle Age	D1	ECHo-1251 (JR 26)	Shrew mandible	515±20	512-547	AD 1403-1438
	Early Middle Age	D3	Poz-81615	Small vertebrate bones	1155±30	1042-1175	AD 775-908

Table 3. Radiocarbon dates (selection) for the Peyrazet, Beg ar Loued and Lano archaeological sites, including those recently obtained in this study from shrew mandibles (shaded). All radiocarbon dates were calibrated using the software Calib Rev 7.0.0 and the Intcal13 calibration curve. "Context" refers to the archaeological layers where the remains were found. The layers follow a chrono-stratigraphical order from bottom to top including an "stratigraphic inversion" for Lano (details given in the Results section). For taxonomical attribution of the shrew mandibles see Table 1.

Area Nr16-21						
Cultural attribution	**Context**	**Lab code**	**Element**	**14C date (BP)**	**2-Sigma cal BP**	**2-Sigma cal BC**
Late Neolithic	d85	Lyon-4418 (SacA-8120)	Charcoal	4705±35	5582-5322	3632-3372
		Lyon-7342 (SacA-20960)	*Cornus* seed	4715±35	5582-5325	3632-3375
	d87	Lyon-7343 (SacA-20961)	Charcoal	4610±30	5460-5146	3510-3196
Middle Neolithic 2	d88	ECHo-1262 (JR 20)	Shrew mandible	5070±40	5912-5728	3962-3778
		Lyon-4407 (SacA-8109)	Charcoal	5110±30	5925-5750	3975-3800
	d92	Lyon-4409 (SacA-8111)	Charcoal	5055±35	5908-5723	3958-3773
	d96	Lyon-4411 (SacA-8113)	Charcoal	5075±35	5911-5742	3961-3792
Middle Neolithic 1	d108	Lyon-4422 (SacA-8124)	Charcoal	5700±35	6620-6406	4670-4456
	d109	Lyon-4423 (SacA-8125)	Charcoal	5645±35	6496-6320	4546-4370
	d110	Lyon-4424 (SacA-8126)	Charcoal	5790±35	6670-6497	4720-4547
	d111	ECHo-1256 (JR 21)	Shrew mandible	5760±40	6659-6454	4709-4504
		Lyon-12108 (SacA-41854)	Charcoal	5790±40	6713-6486	4763-4536
	d115	Lyon-7347 (SacA-20965)	Charcoal	5805±35	6716-6498	4766-4548
Early Neolithic	d140	Lyon-13969 (SacA-49309)	Charcoal	6145±35	7161-6950	5211-5000
		Lyon-11551 (SacA-39068)	Bone	6415±40	7422-7275	5472-5325
	d142	Lyon-11552 (SacA-39069)	Red deer bone	6490±35	7467-7321	5517-5371
		ECHo-1260 (JR 23)	Shrew mandible	6510±40	7492-7323	5542-5373
		Lyon-13970 (SacA-49310)	Charcoal	6865±35	7787-7621	5837-5671
Second Mesolithic	d143	Lyon-13971 (SacA-49311)	Charred hazelnut	6815±35	7698-7587	5748-5637
		ECHo-1247 (JR 24)	Shrew mandible	7130±40	8019-7865	6069-5915
	d147	Lyon-13972 (SacA-49312)	Charred hazelnut	7315±40	8191-8021	6241-6071
		ECHo-1696 (JR 31)	Shrew mandible	7600±50	8536-8334	6586-6384

Area SU16-21						
Cultural attribution	**Context**	**Lab code**	**Element**	**14C date (BP)**	**2-Sigma cal BP**	**2-Sigma cal BC**
Early Neolithic	d22	ECHo-1261 (JR 18)	Shrew mandible	6410±45	7422-7268	5472-5318
Early Neolithic/Second Mesolithic	d26	Beta-282246	Red deer bone	6510±40	7496-7324	5546-5374
	d28	Beta-255118	Red deer bone	6430±50	7428-7269	5478-5319
		Beta-282247	Red deer bone	6490±40	7477-7317	5527-5367
		ECHo-1702 (JR 30)	Shrew mandible	6750±50	7680-7514	5730-5564
Second Mesolithic	d30	Beta-255119	Red deer bone	7310±40	8187-8022	6237-6072
	d34	Beta-282248	Red deer bone	7790±40	8638-8456	6688-6506

Table 4. Radiocarbon dates for the Grande Rivoire archaeological site, including those recently obtained in this study from shrew mandibles (shaded). All radiocarbon dates were calibrated using the software Calib Rev 7.0.0 and the Intcal13 calibration curve. “Context” refers to the arbitrary archaeological layers (d: décapage) where the remains were found. The layers follow a chrono-stratigraphical order from bottom to top in both areas (Nr16-21 and SU16-21). For taxonomical attribution of the shrew mandibles see Table 1.

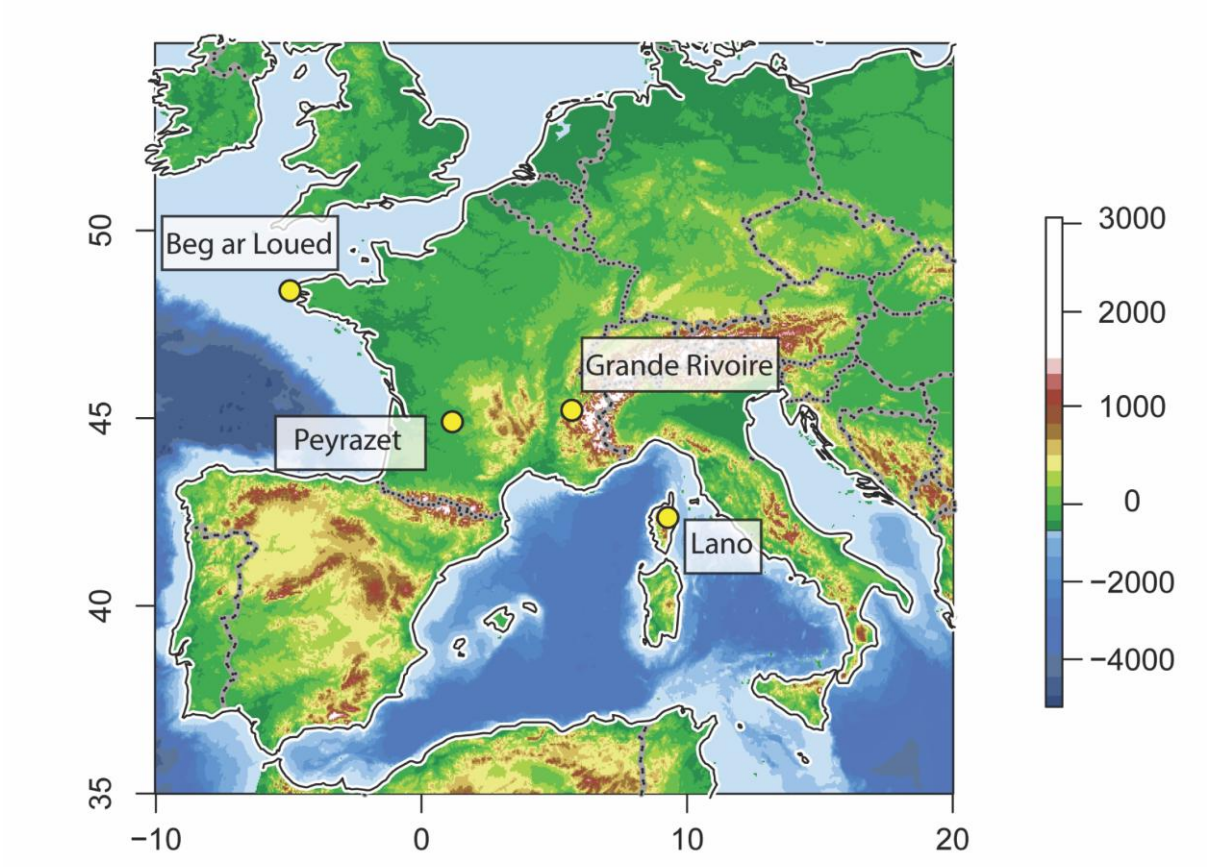

Figure 1. Location of the archaeological sites.

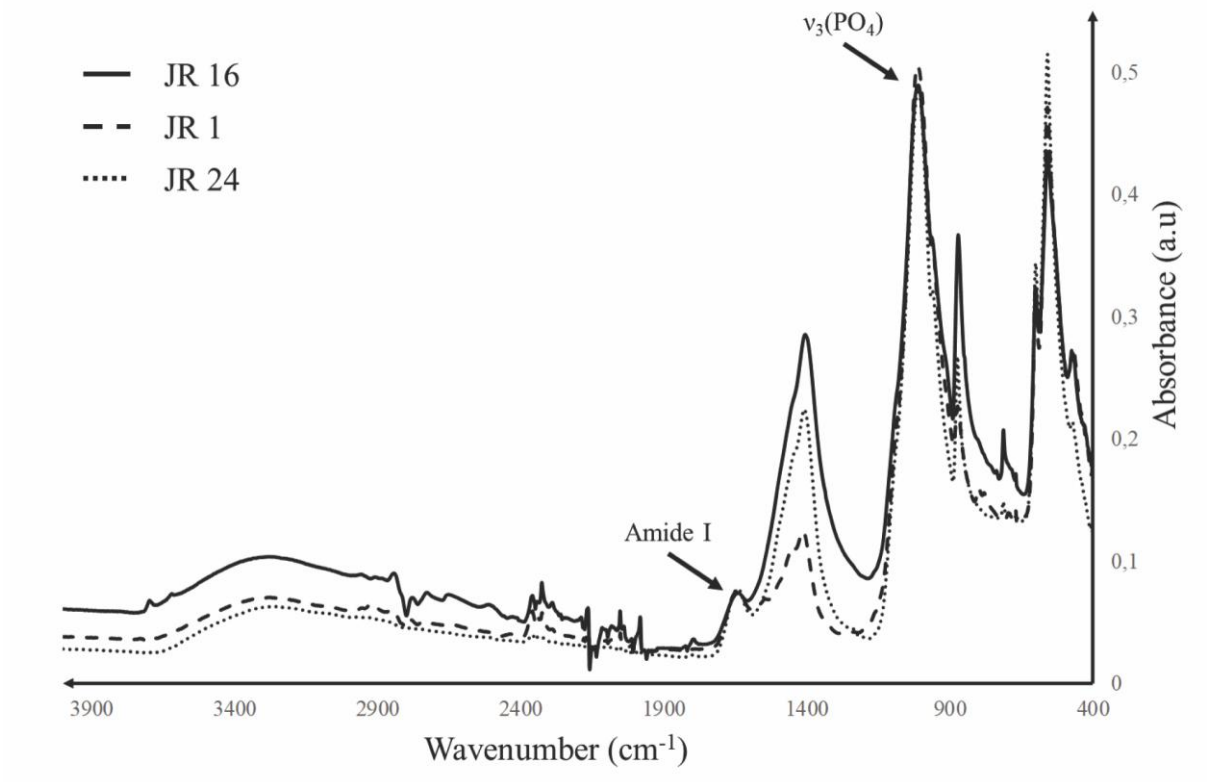

Figure 2. Prescreening ATR-FTIR spectra of selected shrew bone samples: *Neomys anomalus* from Peyrazet (JR 16), *Sorex* gr. *araneus-coronatus* from Grande Rivoire (JR 24) and *Neomys anomalus* from Beg ar Loued (JR 1). The two bands of interest for collagen preservation study [$\nu_3(PO_4)$ mineral and amide I organic bands] are indicated by arrows.

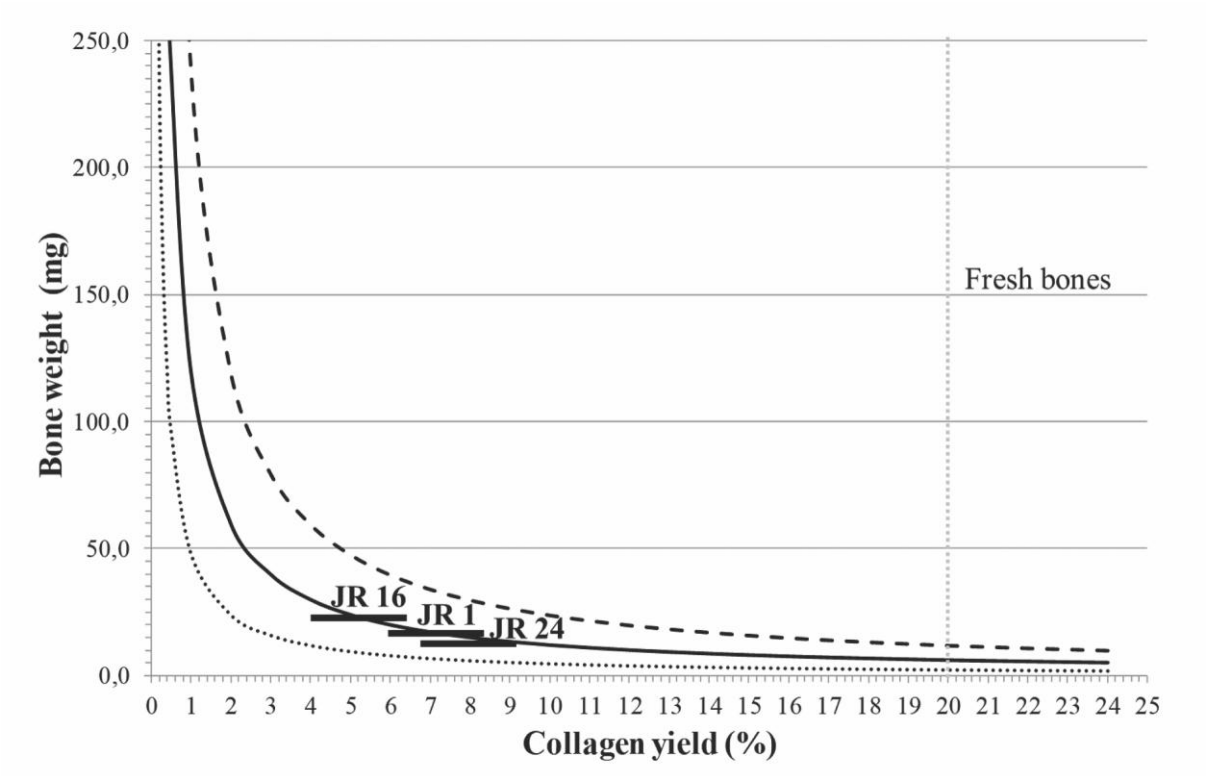

Figure 3. Radiocarbon dating feasibility chart based on the carbon concentration in bone collagen (40%). Y-axis corresponds to the minimum bone weight needed for a reliable radiocarbon date. X-axis corresponds to the collagen content within the bone as estimated by FTIR. Dotted line: to obtain 0.2 mgC. Solid line: 0.5 mgC. Dashed line: conventional 1 mgC. Samples JR 16, JR 1 and JR 24 are reported on the chart, considering the standard error for FTIR estimation (±1.2 %).

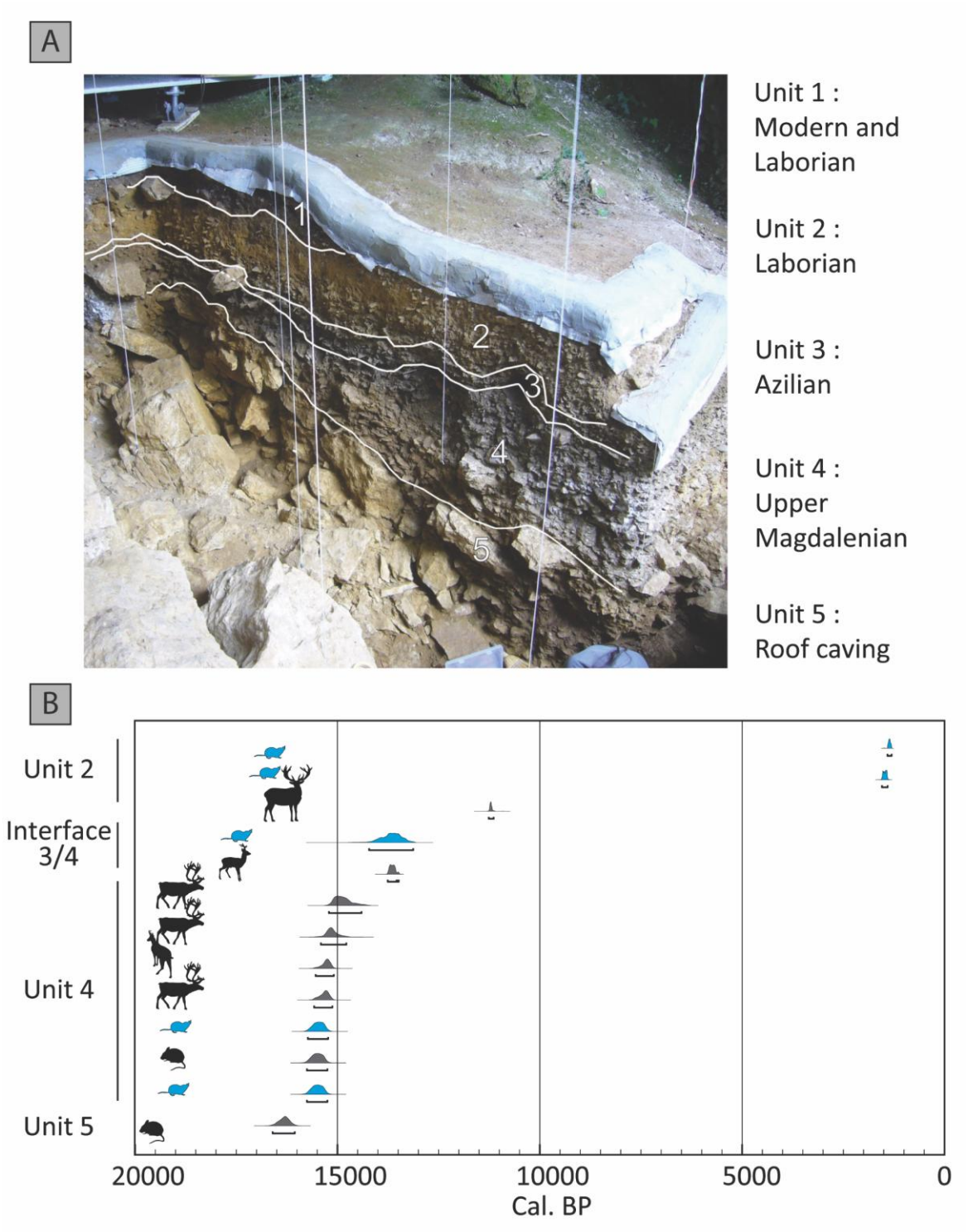

Figure 4. (A) Archaeological profile of Peyrazet showing the chrono-cultural stratigraphic units. (B) Synthesis of the radiocarbon dates discussed in the text, including those newly obtained from shrews (blue) and those from other large and small mammals (black).