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Title: Refining 14C dating of bone >30,000 BP : establishing an accurate chronology for the Middle to Upper Palaeolithic transition in France

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8. Conclusion and Future work

The main focus of this thesis was developing methods for radiocarbon dating bone from archaeological sites dating to the Middle to Upper Palaeolithic transition in Europe, and then applying those methods to obtain dates from a key site from this time period.

The key findings of this thesis are as follows:

Internationally agreed radiocarbon calibration back to 50,000 cal BP

To overcome the ambiguities created by the co-existence of several conflicting ^{14}C datasets beyond 25,000 cal BP the IntCal working group has constructed the calibration curve 'IntCal09' (Reimer, et al., 2009). Earlier problems calibrating samples older than 30,000 cal BP were resolved in 2004 through collaboration between a number of labs and international scientists (including the author of this thesis), which involved discussion about the absolute time scales of the various datasets as well as improvements in the ^{14}C technique itself.

The Radiocarbon dating method is not flawed between 35,000 and 42,000 cal BP

It has been stated by some authors that ^{14}C dating is not possible around 39,000 cal BP because of two key reasons. First, until recently there was a choice of conflicting ^{14}C datasets to calibrate radiocarbon dates in the Palaeolithic age range, which has left room for ambiguity (Mellars, 2006). Second, there have been doubts about the radiocarbon method being capable of producing dates in this time period because of alleged fluctuations of the atmospheric radiocarbon level at this time (Conard and Bolus, 2003, Conard and Bolus, 2008, Fedele, et al., 2008, Giaccio, et al., 2006, Pettitt and Pike, 2001). However, this putative radiocarbon dating anomaly during MUP lasting for millennia simply does not exist. ^{14}C production fluctuations lead to intervals of both accelerated change of radiocarbon years *versus* calendar years and decreased change (i.e., radiocarbon age plateaux), which are well resolved in the current radiocarbon calibration dataset IntCal09. The radiocarbon community has now solved the issues of inconsistent ^{14}C calibration and created a valid calibration curve back to 50,000 cal BP (Reimer, et al., 2009). It is well documented that a geomagnetic minimum (Laschamp Event) and reduction in the circulation in

the North Atlantic (Heinrich event 4) resulted in gradual changes in ^{14}C , but these anomalies are not strong enough to prevent accurate radiocarbon calibration.

Optimising techniques to obtain pure and uncontaminated bone collagen

At the lower age limit of radiocarbon dating, the low ^{14}C activity level and the difficulty of obtaining sufficient and well-preserved collagen, means that bone is an especially challenging material to accurately date. In this thesis I investigated several collagen extraction techniques. I identified a combination of steps which lead to consistent and reliable ages. These procedures combined with the recently installed CO_2 gas collection and graphitization system, enable the department at human evolution at Max Planck-EVA Leipzig, to perform all the required steps in bone dating, from sampling to graphite production for AMS ^{14}C dating.

Well preserved bison and mammoth bones from the North Sea of unknown age were adopted as the long term quality control material, especially to test for background contamination in the sample preparation, with the assumption that these two bones were at least Pleistocene, and ideally older than 50,000 BP. Initial radiocarbon results from two AMS laboratories on collagen prepared at MPI showed the bones to be in the age range of 30,000 to 45,000 ^{14}C BP, but we observed large discrepancies in the radiocarbon ages between different radiocarbon labs. These inconsistencies could have been caused by deficits in the pretreatment methods which we had established for collagen extraction, by insufficient removal of contamination in the samples, in the AMS measurements themselves, or all three.

Therefore we designed a study to investigate the source of these inconsistent dates, and we also compared the results of our pretreatment methods against results from the methods from two of these AMS labs. In this study, as in earlier exercises (Hajdas, et al., 2007, Higham, et al., 2006b, Hüls, et al., 2007), we observed that by using elaborate pretreatment procedures that eliminate both modern laboratory contamination and contamination from degenerated proteins and humic acids we were able to obtain older ages. We still observe discrepancies between the results of different AMS labs, highlighting the many challenges of radiocarbon dating at very low ^{14}C activity.

Accurate chronology of sites covering the transition of Middle to Upper Palaeolithic in France, with a link to climatic events

The radiocarbon dating application in this thesis targeted the site of Les Cottés in France (paper 3 in this thesis).

The nature and duration of the Middle to Upper Palaeolithic transition (MUP) in Western Europe is one of the key ongoing debates in Palaeoanthropology, and it is an area where accurate chronology is essential. Central to this debate is the biological nature of the makers of the different lithic assemblages (Neanderthals and modern humans), and contradictory models have been proposed to explain the cultural evolution of these hominids

Les Cottés is one of the few sites with a complete and well defined sequence covering the Middle to Early Upper Palaeolithic periods in Europe. Refinement of AMS ^{14}C bone dating methods, including ultrafiltration, a new calibration curve (IntCal09, (Reimer, et al., 2009)) and advanced calibration programs (OxCal 4.1, (Bronk Ramsey, 2009)) allow the application of radiocarbon dating to bones from late Middle and Upper Palaeolithic sites in Europe to provide more accurate chronologies for these industries. Radiocarbon dates of 27 bone samples from each archaeological level at this site were obtained. A chronological framework consisting of five phases from the Mousterian to Early Aurignacian periods was created. The results show that the Mousterian and Châtelperronian are contiguous and separated from the overlying Protoaurignacian level by a gap of 1000 years. The fact that a substantial part of the Proto and Early Aurignacian appear contemporaneous, within the resolution of ^{14}C dating, indicates that this transition was rapid in this region. Anatomically Modern Humans are present at the site of Les Cottés at least at 39,500 cal BP, which is roughly coincident with the onset of the strong cold phase Heinrich 4.

Future work

It has been observed in several instances that radiocarbon dates obtained previously from transition period sites could be considered too young, and using more elaborate pretreatment techniques resulted in older ages (e.g. (Higham, 2011, Higham, et al., 2009)). Therefore, it would be useful to revisit these key sites and apply the suite of radiocarbon procedures outlined here. Additionally, we aim to continue dating new sites from this time period with the protocol established here.

