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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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1. Introduction

As observed by Colin Renfrew, radiocarbon has played a revolutionary role in archaeology since the 1950s, first through the invention of scientific dating and second by providing calendar time scales for European prehistory in the Neolithic and Bronze Age periods. More recent examples are the improved constraint of the Eastern Mediterranean Late Bronze Age (Friedrich, et al., 2006), and the redefining of Egyptian chronology (Bruins, 2010, Bruins and Plicht, 2001, Bruins, et al., 2009, Plicht and Bruins, 2001, Ramsey, et al., 2010). However, for the Middle to Upper Palaeolithic (MUP) period in Europe, radiocarbon dating is still controversial. Limited dating evidence and the challenges of radiocarbon dating at the limits of the method mean that there is much room for speculation and controversial conclusions or opinions.

The key questions for this transitional period are if Neanderthals and Modern Humans (MH) overlapped in time, and if so, did they exchange technology, culture or genes, and why did Neanderthals disappear in central Europe at about the time when MH entered this region for the first time?

All these aspects have been widely discussed for several decades and there are numerous strong and divergent opinions. This debate continues mainly because one central element is unresolved, chronology. There are three main aspects which render radiocarbon dating difficult in this time range. This period is close to the limit of the dating range of radiocarbon, so statistical errors can be large. The low remaining ¹⁴C activity makes dating materials very vulnerable to contamination *in situ* and in the lab, and, finally, calibration of radiocarbon ages to calendar ages at this antiquity was not possible until a few years ago.

This thesis addresses these crucial points. Radiocarbon calibration is now possible back to 50,000 cal BP (Reimer, et al., 2009) and moreover, claims of fundamental limitations are not justified (paper 1 in this thesis, Talamo et al. 2012). At the core of this thesis are studies undertaken to establish reliable methods for the extraction of good quality collagen from archaeological bone (paper 2 in this thesis, (Talamo and Richards, 2011)). Based on the experience obtained in these studies, an accurate time frame for key sites, showing the full MUP interval, has been created (paper 3 in this thesis, (Talamo, et al., 2012)).

Chapter 1

This thesis is structured as follows: In chapter 2, the radiocarbon method is outlined. The present state of the calibration of the radiocarbon time scale is presented focusing on the recent extension and consolidation back to the time interval of the MUP. An overview of measurement techniques is given, with an emphasis on AMS as the main radiocarbon measurement technique in use today. The focus of chapter 3 is how to obtain the most reliable ages from bone samples from MUP sites. Bone from archaeological context is the preferred material to obtain dates, especially when compared to charcoal, but it presents challenges due to its open structure. In the past decade it has become apparent that the traditional pretreatment methods are insufficient for very old bone samples, because they may not be capable of removing modern contamination to a satisfactory level. The quantitative aspect of what the addition of a level of modern contaminant ¹⁴C contribution would be is shown and development of new techniques including the use of ultrafiltration is discussed.

In chapter 4 the description of the implementation of the bone pretreatment and sample preparation to produce AMS graphite targets at the Max Planck Institute are outlined. The protocol of lab procedures is presented in detail, including documentation of the production of a database that was created during this thesis.

In chapter 5 (paper 1) the chronology of the Middle to Upper Palaeolithic transition is discussed. Chronology is crucial to the debate about all aspects of technological/cultural contact and exchange between Neanderthals and Anatomically Modern Humans in Europe. Radiocarbon is the backbone of the time frame during this period, even though it is close to the limit of the method because competing dating techniques such as OSL are much less precise or limited in their applicability. However, strong doubts have been raised about the validity of the radiocarbon technique, mainly for the following reasons: 1) Strong fluctuations of atmospheric ¹⁴C have been postulated for the interval from 45 to 35 ka BP rendering radiocarbon ages ambiguous. 2) Until recently several different ¹⁴C datasets were used for calibration in this time period, leaving room for interpretation of the synchronicity of techno-complexes or the role of climate anomalies in human evolution. In this paper it is shown that these issues are now fully resolved, because the large ¹⁴C anomalies are shown to be artefacts beyond plausible physical limits for their magnitude. Previous inconsistencies between ¹⁴C radiocarbon datasets have been resolved and a new radiocarbon calibration curve, IntCal09 (Reimer, et al., 2009) has been created.

Chapter 1

In chapter 6 (paper 2) the crucial steps needed to obtain good quality collagen from ancient bones are studied in detail. Bone is a commonly used material for radiocarbon dating, yet, at ages close to the limit of the method (>30,000 BP), it is a substantial challenge to remove contamination and produce accurate ages. In this paper the preliminary results are reported of a dating study of two bones older than 30,000 years, which were each treated with a suite of pretreatment procedures, including ultrafiltration (Brown, et al., 1988). Substantial differences in the radiocarbon ages were observed, which are most likely linked to crucial steps in the removal of contamination both from the laboratory and from the bone itself.

In chapter 7 (paper 3) the lessons learned are applied to the site of Les Cottés in south-west France, which is one of the rare sites that possesses a complete and well-defined sequence, covering the Middle to Upper Palaeolithic transition period. We undertook an extensive radiocarbon dating program on mammal bone, which allows us to propose a chronological framework of five distinct phases dating from the Mousterian to the Early Aurignacian at this site.

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Chapter 1

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