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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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## 5. Debates over Palaeolithic chronology – the reliability of $^{14}\text{C}$ is confirmed

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### Abstract

The debate about the complex issues of human development during the Middle to Upper Palaeolithic transition period (45-35 ka BP) has been hampered by concerns about the reliability of the radiocarbon dating method. Large  $^{14}\text{C}$  anomalies were postulated and radiocarbon dating was considered flawed. We show here that these issues are no longer relevant, because the large anomalies are artefacts beyond plausible physical limits for their magnitude. Previous inconsistencies between  $^{14}\text{C}$  radiocarbon datasets have been resolved, and a new radiocarbon calibration curve, IntCal09 (Reimer, et al., 2009), was created. Improved procedures for bone collagen extraction and charcoal pretreatment generally result in older ages, consistent with independently dated time markers.

### 1. Introduction

The period of the Middle to Upper Palaeolithic presents one of the major intellectual challenges in archaeology, fuelled by the demise of the Neanderthals and the dispersal of the Anatomically Modern Humans (AMH) in central Europe. Did they ever encounter each other? Did they exchange technology, culture or genes? Is the Neanderthal a forger of the AMH techno-complex, or did AMH invent it independently? Does the Aurignacian reflect the dispersal of the first Modern Humans in Europe? Common to all these questions is chronology, and radiocarbon dating is a central tool to provide this.

Until now, the prospect for a precise Middle to Upper Palaeolithic chronology was controversial in the archaeological world and the reasons are many. First are the intricacies of the radiocarbon method including the requirement of calibration, which has left room for ambiguity (Mellars, 2006). Secondly, there have been doubts about the radiocarbon method being capable of dating this time period because of the alleged extreme fluctuations of the atmospheric radiocarbon level (Conard and Bolus, 2003, Conard and Bolus, 2008, Fedele, et al., 2008, Giaccio, et al., 2006, Pettitt and Pike, 2001). Last but not least, there are the methodological difficulties of using radiocarbon dating close to its limits, with higher errors due to the low remaining  $^{14}\text{C}$  activity of samples from this time period, and resultant vulnerability to contamination *in situ* and in the laboratory. Here the prime dating materials are bone and charcoal; hence the quality of the dates depends strongly on the ability to extract pure collagen from bone and remove traces of contamination from charcoal. This field has seen strong progress over the past two decades. Bone, which is considered closest to the archaeological context, represents a challenge in the extraction of genuine collagen and here the risk of obtaining anomalously young dates is high. Collagen cleaned by ultrafiltration (Brown, et al., 1988) appears to deliver generally older ages, consistent with established time markers such as the Campanian Ignimbrite (CI), but there are still controversial issues

as exemplified by a recent exchange of opinions (Higham, et al., 2006, Hüls, et al., 2007)

We observe that different authors arrive at quite different conclusions regarding the temporal framework of crucial sites and some authors even question the validity of radiocarbon dating. For this reason we consider it useful to revisit some fundamental radiocarbon issues and outline the intense work in the radiocarbon community over the past few years, which has created a solid chain of evidence supporting the use of radiocarbon dating in the time period 30,000 to 45,000 cal BP.<sup>1</sup>

## 2. Special events in the Middle /Upper Palaeolithic time period

The time period prior to 38,000 cal BP experienced strong climate excursions, Dansgaard-Oeschger cycles (DO) 12 to 9 concluding in Heinrich Event 4 (HE 4) (Wang, et al., 2001). Dansgaard-Oeschger cycles are characterized by warm periods ca.1500 years long, with very fast warming (<100 years) and subsequent gradual cooling. The cause could be shifts of the location of the deep water formation in the North Atlantic, i.e. during warm phases the location is further north, near where it is today, whereas during cold phases it is located south of Iceland (Bard, 2002, Rahmstorf and Alley, 2001). Heinrich events are periods in which a greatly increased number of icebergs enter the North Atlantic. The resulting freshwater input may have considerably slowed the major ocean circulation engine, i.e. Meridional Overturning Circulation (MOC) leading to strong cold phases in mid and high latitudes of the Northern hemisphere.

A major volcanic eruption also occurred in this time range; the Campanian Ignimbrite (CI), dated by  $^{40}\text{Ar}/^{39}\text{Ar}$  to 39,395±51 cal BP (Fedele, et al., 2002, Pyle, 1992, Pyle, et al., 2006, Ton-That, et al., 2001). The CI is an important time marker in the Mediterranean and south-eastern Europe. Finally the Laschamp geomagnetic excursion (an interval of extremely low geomagnetic field intensity) is a global stratigraphic tie-point dated to 40,400 ± 1100 cal BP (Guillou, et al., 2004). The potential effect of these events on atmospheric  $^{14}\text{C}$  is presented in detail below.

## 3. Fundamentals of carbon cycle and $^{14}\text{C}$ calibration

$^{14}\text{C}$  dates cannot be used in a straightforward way because the atmospheric radiocarbon level, which determines the initial setting of the ‘clock’, has varied in the past. This is remedied by using  $^{14}\text{C}$  dates of known age material (e.g. tree-rings) to correct, or calibrate, the radiocarbon dates to calendar (cal) ages. The variability of atmospheric  $^{14}\text{C}$  is due primarily to two factors, the first of which is the change in the shielding of the earth against cosmic rays that produce  $^{14}\text{C}$ . The shielding has two components, the Earth's geomagnetic field and the magnetic field in the solar wind. Decreasing magnetic field shielding increases  $^{14}\text{C}$  production and vice versa. The second factor is the distribution of  $^{14}\text{C}$  between global carbon reservoirs. Carbon is primarily exchanged between three reservoirs, the atmosphere, biosphere and ocean. The fraction of carbon in the atmosphere and biosphere is only a few percent compared to the carbon stored in the deep ocean. Deep ocean ventilation (i.e., MOC) controls the distribution of carbon between the atmosphere and ocean, and hence atmospheric  $^{14}\text{C}$

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<sup>1</sup> In this paper radiocarbon ages are denoted  $^{14}\text{C}$  BP, i.e. radiocarbon years before 1950 AD. Calibrated radiocarbon ages are denoted cal BP which are calendar years before 1950 AD and cal BP is used also for calendar ages obtained by other dating methods, such as Ar/Ar or U-Th.

levels. A reduction of MOC would serve to lower the flux of  $^{14}\text{C}$  into the ocean and therefore increase atmospheric  $^{14}\text{C}$ .

The portion of carbon in the biosphere (e.g. plants, soil, sediments and peat) also contributes to atmospheric  $^{14}\text{C}$  variability, but on a much smaller scale than the ocean. To obtain quantitative estimates of the  $^{14}\text{C}$  content of these carbon reservoirs and, most importantly, their temporal development, a number of carbon cycle models have been developed. These models use scenarios of changes in the above-mentioned parameters, including  $^{14}\text{C}$  production or rate of MOC, to calculate the atmospheric  $^{14}\text{C}$  level (Hughen, et al., 2006, Laj, et al., 2002, Siegenthaler, et al., 1980, Stuiver and Braziunas, 1993). In the period of interest, the factors which were the primary contributors to atmospheric  $^{14}\text{C}$  variability are the enhanced production of  $^{14}\text{C}$  (because of the low geomagnetic dipole during the Laschamp Event) and the reduced MOC (due to fresh water input into the North Atlantic during Heinrich event H4).

#### **4. Evidence and discussion of $^{14}\text{C}$ fluctuations in published datasets**

Several  $^{14}\text{C}$  datasets have been taken as evidence for extremely large  $^{14}\text{C}$  fluctuations around 40,000 cal BP – the Bahamas stalagmites (Beck, et al., 2001) and the Tyrrhenian Sea core CT85-5 (Giaccio, et al., 2006) with ages that are systematically too young (ca. 32,000  $^{14}\text{C}$  BP) found in stratigraphical context underlying the CI (ca. 35,000  $^{14}\text{C}$  BP). This latter point appears fully resolved now due to the revised  $^{14}\text{C}$  ages of important Italian sites like “Fumane” in the recent contribution of Higham et al. (2009), where they show that advanced pre-treatment techniques of charcoal lead to  $^{14}\text{C}$  ages in full agreement with the CI age. A recent review of pretreatment of bone and charcoal is given by Ascough et al. (2009) and Higham (2011).

Here we focus on the presence or absence of strong fluctuations of the atmospheric  $^{14}\text{C}$  level, conventionally reported as  $\Delta^{14}\text{C}$ , the deviation from an international standard (Stuiver and Polach, 1977). The CI event coincides with an interval of weak geomagnetic field (Laschamp Event, LE) during which the production of cosmogenic nuclides ( $^{14}\text{C}$ ,  $^{10}\text{Be}$ ,  $^{36}\text{Cl}$ ) was enhanced. For  $^{10}\text{Be}$  and  $^{36}\text{Cl}$ , this effect is seen clearly in the polar ice cores (Beer, et al., 2002), but the signal in radiocarbon is attenuated by buffering through the large ocean reservoir. At the time of its first publication, a Bahamas stalagmite (Beck, et al., 2001) showed a very strong  $\Delta^{14}\text{C}$  spike around 40,000 cal BP which was attributed to a scenario of changes in  $^{14}\text{C}$  production and in carbon reservoirs. However, the strong  $\Delta^{14}\text{C}$  spike in this speleothem record was later determined to be an artefact of an erroneous background correction (Beck, et al., 2008, Hoffmann, et al., 2010). Subsequently, another stalagmite record was obtained from the Bahamas that does not display as strong a signal (Hoffmann, et al., 2008, Hoffmann, et al., 2010), in agreement with other  $^{14}\text{C}$  records (Hughen, et al., 2006).

## 5. Limits to amplitude of large atmospheric $^{14}\text{C}$ fluctuations

The strongest evidence of radiocarbon anomalies comes from the  $^{14}\text{C}$  age sequences of the Tyrrhenian Sea core CT85-5, covered in more detail in chapter 16 of the book: “When Neanderthal and Modern Human met” (Giaccio, et al., 2006). A total of 44 AMS  $^{14}\text{C}$  measurements have been performed on foraminifera which show extremely large  $^{14}\text{C}$  age fluctuations. Within about 800 years the  $^{14}\text{C}$  ages jump from circa 35,000  $^{14}\text{C}$  BP to circa 25,000-20,000  $^{14}\text{C}$  BP and subsequently return to circa 33,000-32,000  $^{14}\text{C}$  BP (Giaccio, et al., 2006). Foraminiferal species and pretreatment have not been specified so it is difficult to evaluate this dataset without further information.

We consider the interpretation of this last dataset as evidence of extremely strong atmospheric  $^{14}\text{C}$  fluctuations as erroneous, primarily because carbon cycle models impose limits to the magnitude and rate of change of atmospheric  $\Delta^{14}\text{C}$ . We imposed extreme scenarios over the characteristic time scales of Heinrich events, DO cycles and the Laschamp Event (ocean ventilation MOC shut off partially or completely, vanishing earth magnetic field, low solar activity) using a previously published carbon cycle model as outlined in the supplementary information, and we obtain a maximum increase of 500 to 1550‰ in  $\Delta^{14}\text{C}$ . These values can be compared to the ranges calculated from the  $^{14}\text{C}$  age drop of the CT85-5 core. The apparent age of 20,000  $^{14}\text{C}$  BP at a true age of 40,000 cal BP corresponds to an atmospheric  $\Delta^{14}\text{C}$  value of more than 9000‰, i.e. more than 6 times the maximum value of the model calculations. Such a high value is completely outside of the range covered by plausible manipulations of the  $^{14}\text{C}$  and the carbon cycle.

The second argument against drastic  $\Delta^{14}\text{C}$  fluctuations is based on the Cariaco Basin  $^{14}\text{C}$  dataset, which has several data points at 150-200 year sampling resolution in the age range of the CI (Hughen, et al., 2006), but does not show unusually large fluctuations.  $\Delta^{14}\text{C}$  in Cariaco has a peak of up to 700‰ between 44,000 and 36,000 cal BP (Hughen, et al., 2006) changing smoothly in this interval, mainly caused by the low geomagnetic field during LE. While the marine radiocarbon reservoir will no doubt attenuate a peak in atmospheric  $^{14}\text{C}$  to a certain extent, depending on the rate of change of  $^{14}\text{C}$ , we know from nuclear weapons testing measurements that such a large attenuation as that required to support a 9000‰ value for the atmosphere could not have occurred, even for decadal scale or shorter anomalies. In fact, the longer centennial-to-millennial time scales of the purported  $^{14}\text{C}$  anomaly at 40,000 cal BP would result in the marine signal being in near equilibrium to the atmosphere.

The observed gradual changes in  $\Delta^{14}\text{C}$  do not invalidate radiocarbon dating at all, because the calibration procedure is designed to account for these anomalies (Fig. 1). We conclude that the  $^{14}\text{C}$  age inversion of up to 15,000  $^{14}\text{C}$  years in the Tyrrhenian Sea core could not have been caused by fluctuations of the atmospheric  $^{14}\text{C}$  level.

## 6. Calibration of radiocarbon dates by IntCal09 back to 50,000 cal BP

Due to new data and the application of stringent quality criteria, the previous limitation for calibration of  $^{14}\text{C}$  dates (26,000 cal BP, IntCal04 (Reimer, et al., 2004)) no longer exists. After four years of intense discussion and review of new datasets the IntCal Working Group created an extension back to 50,000 cal BP, IntCal09 (Reimer, et al., 2009) which was approved by the radiocarbon community. Using this data set, reliable and statistically robust calibrated ages close to the limit of the method can be obtained. While the IntCal Working Group will continue to make refinements to the radiocarbon calibration curve, particularly with regard to marine reservoir ages, there are unlikely to be substantial changes in this time period.

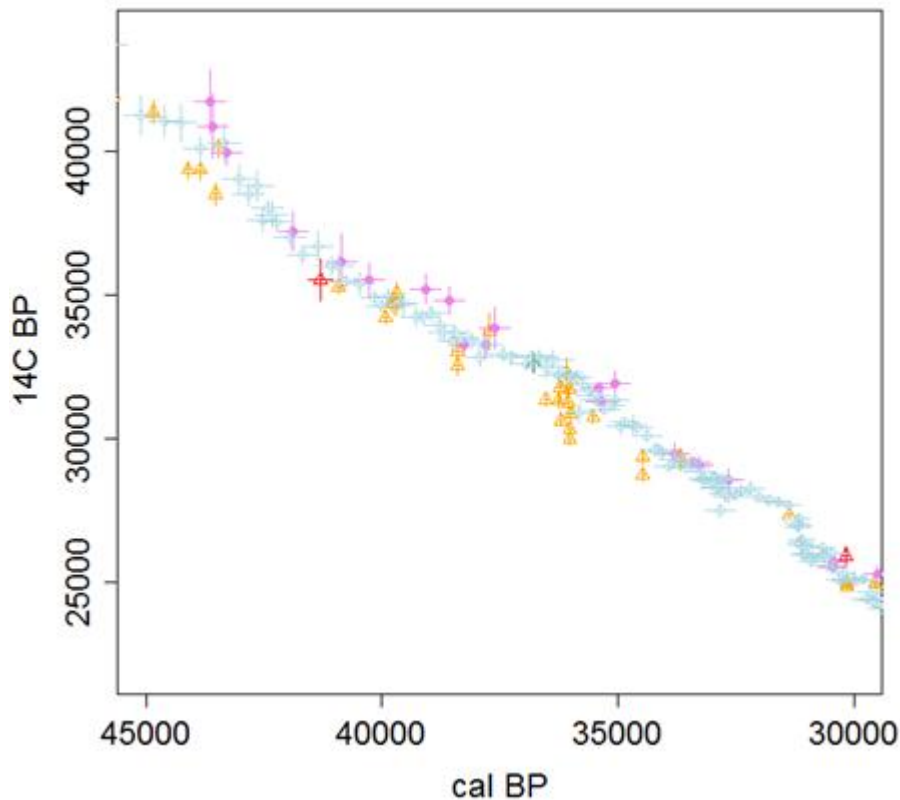
## 7. Conclusions

The unusual sequence of events during Middle to Upper Palaeolithic leaves ample room for speculation and competing theories, even more so when a rigid age control is lacking. At least for chronological issues, we can provide a remedy. The putative radiocarbon dating anomaly during Middle to Upper Palaeolithic lasting for millennia does not exist.  $^{14}\text{C}$  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 solved the issues of inconsistent  $^{14}\text{C}$  datasets and created a valid calibration curve back to 50,000 BP (Reimer, et al., 2009). Improved protocols for bone collagen extraction and charcoal pretreatment result in calibrated ages in agreement with the CI time marker.

$^{14}\text{C}$  dating of samples older than 30,000 years is still challenging and requires outstanding efforts in sample selection and in laboratory procedures. Published dates need to be critically assessed for being too young due to incomplete decontamination, before they are incorporated into archaeological concepts. We see a bright future for radiocarbon dating even at ages close to the limit of the method.

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*Fig. 1:  $^{14}\text{C}$  calibration data in the interval 30,000 to 45,000 cal BP, Cariaco (Hughen, et al., 2006, light blue), Corals (Fairbanks, et al., 2005, orange), Iberian margin marine sediments (Bard, et al., 2004, pink), Corals (Bard, et al., 2004, red), Corals (Cutler, et al., 2004, dark green).*

### **Supplementary information**

If we consider a reduction of the geomagnetic field intensity to zero (maximum case) lasting for 1000 years (Laj, et al., 2002), the atmospheric  $\Delta^{14}\text{C}$  increases by  $\sim 420\text{‰}$  as calculated by carbon cycle models. For a reduction of the geomagnetic field lasting 2000 years, the  $\Delta^{14}\text{C}$  increase is only slightly more ( $\sim 550\text{‰}$ ). Another scenario could be the reduction in MOC by 30%; this causes atmospheric  $\Delta^{14}\text{C}$  to increase by 40‰, in agreement with Delaygue et al. (Delaygue, et al., 2003) and a reduction of MOC by 50% results in an atmospheric  $\Delta^{14}\text{C}$  increase of 85‰. The simultaneous reduction in geomagnetic field and MOC (lowering geomagnetic field intensity to 0 and MOC by 30% for 1000 years) yields an atmospheric  $\Delta^{14}\text{C}$  increase of 500‰ (650‰ if the MOC is reduced by 50%). The most extreme scenario would be reducing both geomagnetic field intensity and solar activity to 0 for 1000 years, together with a 50% reduction in MOC, resulting in an atmospheric  $\Delta^{14}\text{C}$  increase of 1550‰.

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