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# Summary

As observed by Colin Renfrew radiocarbon dating has had a revolutionary role in archaeology since the 1950s. The first contribution made by radiocarbon was to produce accurate direct dates for archaeological materials. The second main contribution was to provide calendar time scales for European prehistory, especially starting from the 1970s for the Neolithic and later periods. Today we are experiencing a third key contribution of radiocarbon dating, as it is being used to attain accurate chronologies for prehistoric periods close to the limit of the method, such as the transition from the Middle to Upper Palaeolithic.

## Radiocarbon basics

$^{14}C$ is created in the upper atmosphere and enters the global carbon reservoirs mainly as $^{14}CO_2$ in the atmosphere. Through photosynthesis $^{14}C$ enters plants and ultimately all living organisms. After the death of an organism, exchanges with the carbon reservoir no longer take place and $^{14}C$ decreases by decay to $^{14}N$ at a known rate (half-life). Measuring the amount of remaining $^{14}C$ in the remains of dead organisms is the basis of the radiocarbon method.

As there have been fluctuations in the production of $^{14}C$ in the past, it is necessary to calibrate radiocarbon ages to calendar ages. Radiocarbon ages are converted to calendar years by means of calibration curves based on independently dated organic remains, such as tree-rings, marine corals or lake and marine sediments. In 2009 the IntCal working group constructed a new calibration curve spanning back to 50,000 cal BP, a development of key importance for this thesis. The radiocarbon dates presented in this thesis have been measured using the Accelerator Mass Spectrometry (AMS) technique. AMS is used to detect the different isotopes of carbon ($^{12}C$, $^{13}C$, $^{14}C$) by separating them according to their respective mass.

## Radiocarbon calibration around 40,000 years cal BP

The Middle to Upper Palaeolithic transition is the period during which Neanderthals disappeared and modern humans made their first appearance in Europe. Radiocarbon dates on organic remains from late Middle and early Upper Palaeolithic sites and their calibration have proven especially controversial. Several $^{14}C$ datasets have been taken as evidence for extremely large $^{14}C$ fluctuations around 40,000 cal BP, such as the set of dates from the Tyrrhenian Sea core CT85-5, which if taken at face value indicate strong radiocarbon anomalies at that point in time. The sequence of dates from the above-mentioned Tyrrhenian Sea core appears to suggest that within an 800 year period the $^{14}C$ ages fluctuated from circa 35,000 $^{14}C$ yr BP to circa 25,000-20,000 $^{14}C$ yr BP and then back to circa 33,000-32,000 $^{14}C$ yr BP. I consider the interpretation of this dataset as erroneous, because a $^{14}C$ age inversion of up to 15,000 $^{14}C$ years could not have been caused by fluctuations in the level of atmospheric $^{14}C$. The enhanced $^{14}C$ production during the magnetic low of the Laschamp Event is well documented in high resolution $^{14}C$ records, such as Cariaco, which provide a solid basis for radiocarbon calibration for this time period.

## Bone pretreatment

Despite the considerable potential offered by bone collagen for radiocarbon dating, dating bones can be problematic as these organic materials are often degraded and can be subject to contamination in archaeological contexts. The effect of contaminating carbon on the radiocarbon ages is more severe the older the bone used for dating. As an indicator of contamination and/or degradation of collagen different authors use C:N ratios, $\delta^{13}C$ and $\delta^{15}N$ values, and amino acid composition. It is generally assumed that contamination is likely to have occurred when atomic C:N ratios fall outside the range observed for modern animals and humans.

I investigated a range of different pretreatment techniques and methods of cleaning the molecular weight (MW) separation filters and pre-filter elements. For these experiments I used bones of a mammoth and a bison recovered in prehistoric deposits from the North Sea and I sent the same collagen extracts from these specimens to three AMS facilities. The results varied considerably, between 31,660 and 35,280 $^{14}C$ BP in the case of the mammoth and between 40,200 and 47,300 $^{14}C$ BP in the case of the bison. Most of this variability could be explained by the different pretreatment methods employed and by differences in measurements between the three AMS facilities. As a result of these experiments, I devised an optimal pretreatment protocol, which produces consistent age estimates and results in low intra-sample age variability. This pretreatment method is the protocol now in use at the MPI-EVA.

## Sequence of bone preparation for radiocarbon dating at MPI

All the bone samples presented in this thesis were subject to the following pretreatment procedures, usually in batches of up to 12 samples:

- Entry in database
- Pulverisation of bone

- Decalcification
- Removal of humics
- Gelatinization
- Cleaning of the filters and checking for the removal of contamination
- Ultrafiltration
- Freeze drying

All the collagen obtained after the pretreatment outlined above is graphitized according to the following procedures:

- Loading of collagen into tin caps
- Combustion in an Elemental Analyser (EA)
- Determination of carbon yield and C:N ratio
- Determination of $\delta^{13}C$ and $\delta^{15}N$ in a mass spectrometer
- Cleaning the $CO_2$ gas containers and conditioning of the iron catalyst
- Collection of $CO_2$ in the rigs
- Addition of hydrogen
- Conversion of $CO_2$ into graphite in the graphitizer
- Check of graphitization parameters
- Preparation of blank samples
- Preparation of shipment to an AMS facility and submission to the AMS laboratory for radiocarbon measurement.

## Radiocarbon chronology of the Middle to Upper Palaeolithic site of Les Cottés

The pretreatment methods developed in the present thesis were applied to bone samples from the Palaeolithic site of Les Cottés (France). The cultural levels investigated span from the Mousterian to the Aurignacian, and include the Châtelperronian. These cultural phases were correlated to episodes of rapid climatic change which occurred from around 47,000 to 35,000 cal BP. During this period there were 6 warmer stages called Dansgaard-Oeschger (DO) events, and the marked cooling episode known as Heinrich event 4 (H4). The results of the AMS radiocarbon dating program on bone collagen samples from Les Cottés have allowed the development of a chronological framework that is coherent with the archaeological stratigraphy of the site.

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. Radiocarbon dates have been obtained for 27 bone samples from each archaeological level and grouped on the basis of the five cultural phases present at the site, from the Mousterian to the Early Aurignacian. The Mousterian and Châtelperronian are separated from the overlying Protoaurignacian level by a gap of approximately 1000 calendar years. The internal temporal relation between the Mousterian and Châtelperronian is not fully resolved by our dates, this aspect will be addressed by future work at the site. 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 the region in question. Anatomically Modern Humans were present at the site of Les Cottés at least by 39,500 cal BP, roughly coincident with the onset of the markedly cold phase of Heinrich event 4.