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Radiocarbon and mammoth bones: What's in a date

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ABSTRACT

The Radiocarbon dating method has developed into a reliable dating method for organic sample materials. The latest calibration curve released enables numerical dating covering the complete dating range of the method. However, the category of fossil bones is the subject of discussion concerning validity of the dates, in particular for the oldest part of the ¹⁴C timescale. This is a complex interplay of sample material integrity, contamination issues and proper blanks. In practice, a safe upper limit for ¹⁴C dates of bone material appears to be 45,000 BP (50,000 calBP).

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1. Introduction

The Radiocarbon (¹⁴C) dating method has contributed significantly to many disciplines studying the past, as it provides a physical yardstick of time (Libby, 1952). Organic samples such as charcoal, bone, wood, peat, shells, and plant remains can be directly dated to about 50,000 years ago. Since the conception of the method in the 1950s, it has continuously been improved; the most significant improvements being the introduction of AMS (Accelerator Mass Spectrometry) enabling small sample analysis (Tuniz et al., 1998), and the establishment of a calibration curve for the complete dating range (Reimer et al., 2013).

Many chronological questions have been solved by Radiocarbon dating over the years. However there always have been debates concerning the acceptance of ¹⁴C dates, in particular in archaeology (Renfrew, 1999). These continue to the present day – witness, to mention just one prime example, the dating of the Santorini/Thera volcanic explosion (Antiquity, 2014).

In this contribution, the focus will be mainly on a specific category of ¹⁴C dating most relevant for paleontologists researching Late Pleistocene mammal remains: bones, in particular from its most iconic representative, the woolly mammoth. Also here are sometimes vehement discussions concerning validity of dates, sample quality and methodology. This is triggered by relatively recent developments of the ¹⁴C method, in particular the

introduction of AMS and sample treatment improvements including the ultrafilter method. The latter was coined a “revolution for Palaeolithic archaeology” (Mellars, 2006) but is the subject of debate (Hüls et al., 2009) and to extensive testing using palaeontological bone (e.g., Fiedel et al., 2013).

It is important to note that bone is perhaps the most difficult (or sensitive) material to date by ¹⁴C, in comparison with for example charcoal or wood. The literature is polluted by many invalid bone dates; the older the samples, the worse this becomes (e.g., Graf, 2009; Vartanyan, 2013).

There is a variety of parameters determining the outcome of ¹⁴C dating (correct or wrong) of fossil bone, easily causing confusion. There are good bones and bad bones (in terms of sample quality), and there are good measurements and bad measurements (in terms of ¹⁴C laboratories). However, there is not a simple one-to-one correlation between these.

The danger of circular reasoning is present. When an improvement in the method produces dates that fit expectations of the user (usually in the older direction), that does not necessarily mean that the dates are then correct. Also, when dates are different from expectations (usually in the younger direction), that does not automatically mean they are wrong.

When two independent age assessments are not consistent with each other and there is no obvious objective reason or solution, then all we can say is: at least one of them must be wrong. The purpose of this contribution is to review present knowledge and enable the ¹⁴C user community to be better able to judge the validity of bone dates, in particular near the detection limit of the method.

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2. The Radiocarbon timescale

Originally (during the early 1950s), ^{14}C dates were reported in BP (Before Present), just as it is common practice in other dating techniques, most notably in the earth sciences. Early dates were significant, often revolutionary, but crude with 1-sigma errors often a few hundred BP (Libby, 1952). The radioactivity was measured relative to a standard corresponding to values of the “present day”, 1950 at the time, which is a chemical substance called oxalic acid. The half-life value of 5568 years, as determined by Libby was used.

It was soon discovered that there were problems with both. Modern values appeared to have been changed because of fossil fuels (which do not contain ^{14}C), so that the 1950 oxalic acid has 5% less ^{14}C than the natural value before the anthropogenic effects, affecting atmospheric CO_2 (and its isotopic values). Also, de Vries (1958) discovered that significant natural variations occur in the atmospheric ^{14}C content. These are caused by a changing cosmic ray flux which produces the cosmogenic isotopes such as ^{14}C . Further, the half-life has later been accurately determined as 5730 years. Finally, mass dependent effects (isotope fractionation) were discovered which influence the ^{14}C content of a sample (and thus their age).

In order to solve these problems the ^{14}C laboratories have agreed to the following convention:

- 1). The ^{14}C activity (i.e. the $^{14}\text{C}/^{12}\text{C}$ ratio) is measured relative to that of an international standard (oxalic acid). The value of this reference is standardized to a specific activity and reference year (1950)
- 2). It is corrected for fractionation using the $^{13}\text{C}/^{12}\text{C}$ ratio of the sample to an agreed standard value
- 3). The ^{14}C age is calculated using the original half-life (5568 years)
- 4). The ^{14}C age is reported in the unit “BP”.

Thus, the ^{14}C timescale is defined. Note that this timescale is “elastic” because of natural variations in the ^{14}C content of nature. The defined ^{14}C timescale needs to be connected to the calendar timescale by calibration. This calibration automatically takes into

account natural ^{14}C variations and the half-life uncertainty. The only uneasy element in this definition is BP, which does not mean Before Present in the literal sense. However, the use of this term has been so widespread that all attempts to change it have failed.

Calibration of the ^{14}C timescale is possible by measuring ^{14}C in tree rings, which are dated absolutely by dendrochronology. This is presently possible back to about 12,500 years ago (Friedrich et al., 2004).

Only recently, calibration curves became available covering the complete Radiocarbon dating range of 50,000 years (Reimer et al., 2013). This calibration curve Intcal13 is shown in Fig. 1. The older part of the curve is derived from U-series dated corals and foraminifera, and from laminated sediment from Lake Suigetsu, Japan (Bronk Ramsey et al., 2012). The ^{14}C ages are shown in BP, and the calendar timescale in calBP. The latter is defined as years relative to 1950 AD, i.e. calBP = 1950 – AD (Mook and van der Plicht, 1999).

3. ^{14}C methodology

3.1. Sample treatment

Before the actual ^{14}C measurement, sample materials have to be chemically pretreated in order to isolate the datable fraction, and to remove contaminants (Mook and Streurman, 1983). Bone dating proved to be difficult in the early days of Radiocarbon. Dating of “bulk” carbon was practiced, often giving young ages. Bone samples were originally not even listed among sample materials to be used (Olsson, 2009). Sometimes, the dating of bone apatite was successful. However, secondary calcite from the burial environment can infiltrate the bone. This obviously hampers bone ^{14}C dating based on the inorganic fraction, which must be based on primary (biogenic) and not secondary (diagenetic) carbonate. Longin (1971) therefore developed a collagen extraction technique, enabling ^{14}C dating of the organic bone component. Collagen does not exchange carbon with the environment. This therefore has become the main dating tool for bone. Nevertheless, carbonate dating usually gives good dates for teeth and tusks, simply because they are more resistant to degradation and less sensitive to exchange of Carbon with the environment.

The main quality parameters for bone collagen isotopic analysis are the Carbon and Nitrogen content. Their values should be higher than ~30% and 12%, respectively, while the acceptable range for the C/N ratio (normalized for the atomic mass ratio 14/12) is 2.9–3.6 (DeNiro, 1985; van Klinken, 1999). These values are based on those of fresh animal bone.

Additional collagen quality information is provided by the stable isotopes ^{13}C and ^{15}N . Their numbers are expressed in so-called delta values $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$, which are defined as: $\delta X = (R_{\text{sample}}/R_{\text{standard}}) - 1 \times 1000\text{‰}$, where X stands for ^{13}C or ^{15}N and R stands for $^{13}\text{C}/^{12}\text{C}$ or $^{15}\text{N}/^{14}\text{N}$.

For bone collagen, the $\delta^{13}\text{C}$ values are generally in the range –18 to –22‰ (Mook and Streurman, 1983). Impurities generally result in lower $\delta^{13}\text{C}$ values, as the insoluble compounds have $\delta^{13}\text{C}$ values of –22 to –29‰. Note that the stable isotope ratios $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ for bone collagen also depend on the food source of the organism (Kohn, 1999). This is not further discussed here.

Bones which are degraded show deviating collagen quality parameters. Their dating results are often questionable. Usually they are too young, which must be caused by modern contamination apparently not removed by the collagen preparation procedure. For this reason, the Oxford ^{14}C laboratory has developed the ultrafilter method. Ultrafiltration is used to purify the collagen, separating out the smaller and lower molecular weight fractions which seem to have been the major source of more modern organic contaminants

Fig. 1. The calibration curve Intcal13 (Reimer et al., 2013), showing the relation between Radiocarbon years (BP, vertical) and calendar years (calBP, horizontal).

(Bronk Ramsey et al., 2004). Impossibly young dates became significantly older after using the ultrafiltration method (e.g., Jacobi et al., 2006).

However, extensive testing by the Kiel ^{14}C laboratory showed that filtration does not necessarily produce a better date (Hüls et al., 2009). Also, as in other pretreatment steps of the collagen material prior to analysis, there is a risk that the filters themselves introduce contamination to the sample material, instead of only removing it (Minami et al., 2013).

In a collaborative effort by the laboratories of Oxford and Groningen the dating results of the same bone materials, with (Oxford) and without (Groningen) ultrafiltration were compared. This way, Neanderthal bones from various locations were dated. The bones were well preserved, yielded good quality collagen, and there were apparently no additional contaminants removed by ultrafiltration. Both methods show consistent results, ranging between 32 and 36 ka BP (Semal et al., 2009; Crevecoeur et al., 2010; Maroto et al., 2012). For Pleistocene large mammal bone, similar tests are underway for the brick quarry site Coenen in Germany by the 3 laboratories in Groningen, Kiel and Oxford (Matzerath et al., 2012). Hence, the standard used chemical pretreatment of bone material works in general quite well for well preserved bones. The addition of ultrafiltration to the standard chemical pretreatment procedure seems to work well for collagen from bone material that has not been well preserved and is contaminated with carbon from other sources.

3.2. Measurement of ^{14}C

Most AMS systems measure the ^{14}C contents in samples in the form of graphite. Therefore, after the pretreatment, a small amount (approximately 6 mg) of collagen is combusted into CO_2 gas, which is subsequently converted into graphite by a reaction with H_2 (Aerts-Bijma et al., 2001).

The AMS measures the $^{14}\text{C}/^{12}\text{C}$ and $^{13}\text{C}/^{12}\text{C}$ ratios in the graphite (van der Plicht et al., 2000). These ratios are converted into ^{14}C activities, $^{14}\text{a}_\text{N}$ (see Mook and van der Plicht, 1999). This is the radioactivity, measured relative to a standard and ranges between 0% (no activity) and 100% (activity of the standard). To obtain this activity value, $^{14}\text{a}_\text{N}$, the measured $^{14}\text{C}/^{12}\text{C}$ ratio of the sample is corrected for isotope fractionation and it is corrected for the $^{14}\text{C}/^{12}\text{C}$ ratio that was measured in a known infinitely old sample ('background sample'). Infinitely old samples, which do not contain a measurable amount of ^{14}C anymore, show the amount of ^{14}C that has been added to the sample during preservation, sampling, (chemical) pretreatment and/or analysis. Very old and ^{14}C -free materials always yield detectable $^{14}\text{C}/^{12}\text{C}$ ratios, because at least both the chemical treatment and physical measuring processes cannot be done completely ^{14}C free. Thus, background sample materials are generally used to correct for the contribution of ^{14}C -containing carbon contaminations in the investigated material (collagen in case of bone samples). The ^{14}C ages are calculated from the obtained activity by the exponential decay law of radioactivity, using the conventional half-life.

Apart from these technical aspects of measuring ^{14}C in the laboratory, one has to be aware of what this ^{14}C measurement of a particular sample actually represents.

This makes that, from the users point of view, the following questions have to be considered:

- what is the ^{14}C event for the sample material;
- how is the ^{14}C event associated with the event of interest (e.g., the human event);
- does the material for which the ^{14}C event has been identified meet the requirements for a conventional ^{14}C age?

For a thorough discussion of these issues we refer to van Strydonck et al. (1999), and to an earlier publication by Mook and Waterbolk (1985).

A final quality check of dates concerns laboratory intercomparisons. The ^{14}C laboratories check their performances (reliability and reproducibility) by all measuring the same sample. The most recent one published is known as VIRI (Fifth International Radiocarbon Intercomparison). The results of this intercomparison (as well as earlier ones) can be found in the literature (e.g., Scott, 2003) and on www.radiocarbon.org. This includes fossil bone samples, mostly of Holocene age.

At present, there is a new intercomparison (known as SIRI: the Sixth International Radiocarbon Intercomparison) including Pleistocene bones. This will be discussed below.

4. The oldest part of the ^{14}C timescale

4.1. General

The natural concentration of cosmogenic isotopes such as ^{14}C is extremely small. The relative $^{14}\text{C}/^{12}\text{C}$ content in modern material is 10^{-12} ; for 50,000 year old samples, this is decreased to $\sim 10^{-15}$. Such low concentrations make the method very sensitive for allochthonous carbon containing ^{14}C . This is contamination, usually making the samples date too young when not adequately removed or corrected for. Potential problems with ^{14}C dates (in particular bone) are in general "amplified" towards the oldest part of the dating range, beyond 35,000 BP (^{14}C activities $^{14}\text{a}_\text{N} < 1.5\%$). A 45,000 year old sample will be measured as 35,000 BP, when there is 1% modern contamination in the dated material (Mook and Waterbolk, 1985). Compared with the conventional radiometry technique, AMS is more sensitive for contamination during the pretreatment of the sample because the sample size is a factor of 1000 smaller (Lanting and van der Plicht, 1993/1994).

If this contamination had entered bone materials during preservation, conservation (glue) and sampling, different pretreatment steps can be used to remove these contaminants. However, the rate of success in separating the carbon of interest from the contaminating carbon depends on different factors (rate and type of degradation, the type of intrusion of foreign carbon, different methods for removal of contaminants). The analyzed graphite can still contain some remaining carbon from other sources than the material of interest. During the pretreatment steps, foreign carbon can be added to the sample as well. Although the procedures of the method should be such that the contamination risks during the pretreatment and measurement are always as low as possible, the contributions are usually still measurable and should therefore be quantified and corrected for in the measured ^{14}C results using background sample material.

The necessary correction for carbon contaminations in the investigated carbon material introduces uncertainties in the activity value that is used to calculate the age of the unknown sample. It therefore also introduces uncertainties in this calculated age. This is especially the case for very old unknown samples, as the measured ^{14}C signal of these samples is, similar to the background samples, is mainly determined by contamination. Dating problems occur if the measured background ^{14}C signal ($^{14}\text{C}/^{12}\text{C}$ ratio in case of AMS), is not representative for the ^{14}C signal of the contamination in the unknown sample and/or has a variability that exceeds the net $^{14}\text{C}/^{12}\text{C}$ ratio of the unknown sample after background correction.

4.2. Age limit

The practical age limit of dated bones is largely determined by proper removal of contamination and by the quantification and

correction of remaining contamination based on the analysis of background samples. The usual material that is used as background sample to correct for the addition of ^{14}C containing carbon during the chemical pretreatment, combustion, graphitisation and AMS measurement of the sample, is anthracite of geological age. The use of anthracite as background material works well for the vast majority of dated samples. It is representative for wood, plants, charcoal and other charred remains. However, for bones these kinds of samples are not necessarily representative background samples, because, besides of the differences in the material structures, the chemical pretreatment of bones is very different compared to the other mentioned materials.

For interpretation, using other material than bone as background sample can have severe consequences. For example, a bone can yield a date of 47,000 BP when charcoal is taken as the background sample, while in reality its ^{14}C amount is beyond the detection limit (and its age can thus also be, for example, 100,000 years). In this case the background sample could not correct for all the contamination that was still in the investigated sample material. This contamination either was not removed well enough during the pretreatment, or more contamination had been added to the collagen sample than to the charcoal background sample during the analysis work.

Such effects influence distributions of mammoth dates, as shown for Alaska (Matheus et al., 2003). The distribution shows a peak around 35,000 BP which can very well be an artefact caused by background correction, using samples that were not representative for the investigated bone material.

Then why not take infinitely old bone as background material? This only works for good quality old bones, and has been investigated thoroughly by Cook et al. (2012).

Bones from permafrost usually have good quality to serve as background, because they are very well preserved. A prime example is the Arilakh mammoth. A large piece of bone could be dated in Groningen to beyond 50 ka BP, employing both radiometric and AMS methods. For details, refer to Mol et al. (2006).

Could such bones used as background material? It does provide a good background for the chemical treatment in the laboratory. But it is not representative for contamination/degradation processes of bones not from permafrost.

A special background study was performed for Schöningen, a well known Pleistocene site in Germany, about 90 km east of Hannover. It is an opencast browncoal mine that revealed ample paleontological and palaeolithic objects. Most spectacular is the find of 8 wooden spears with an age of 300–400,000 years (Thieme, 2007). Thousands of faunal bones were collected over the years. The structural quality of some of the bones appeared very good, inspiring us to test for the presence of collagen, with positive result. This spawned an investigation of the stable isotopes ^{13}C and ^{15}N in collagen from selected bones – including Elephantidae (Kuitens et al., Accepted for publication). Because of their age, the bones are ideal for background tests for the ^{14}C dating method as well.

As expected, 5 selected bone samples (all from horses) showed dates at background level. The measured activities and resulting ^{14}C dates are shown in Fig. 2. Also shown is a mammoth bone from Latton Quarry (UK), which is known to date from MIS 7 (Cook et al., 2012), showing infinite age as well. Note that the anthracite blanks are subtracted for the analysis shown. Negative net activities correspond to dates older than the background (anthracite) subtracted.

The reported age limit of dating results is, beside correct background corrections, also determined by calculation limits and a convention. The ^{14}C ages are calculated from the measured radioactivities. Both activities and ^{14}C ages are shown in Fig. 2. For ^{14}C ages near the detection limit, the errors in BP become asymmetric,

Fig. 2. Illustrative plot showing the ^{14}C measurements of bone material known to be background, and bones from the North Sea region (one mammoth selected for SIRS, and 9 *Elephas antiquus* bones). Shown are the ^{14}C activities ($^{14}\text{a}_\text{N}$) in % (left vertical axis), and the corresponding ^{14}C ages in BP (right vertical axis). On the right, 4 standard values (blue labels) are plotted: the modern reference (off scale), and 3 typical backgrounds. The arrow indicates a “borderline” *Elephas* date (discussed in the text). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

as is apparent for the various old dates discussed above. Also, error ranges can include negative activities. Mathematically, negative activities would correspond to ages older than infinite, which is impossible. Therefore the ^{14}C community adopted a convention for such “old ages” (Olsson, 1989).

The convention is based on the measurement error sigma; when the measured activity becomes smaller than 2 times its error, the age is given as this limit (the so-called 2 sigma criterion). For a detailed explanation, refer to Olsson (1989) and van der Plicht and Hogg (2006).

4.3. Case study discussion: the North Sea

The North Sea is well known for its unique finds of Late Pleistocene mammal fossils – among which are mammoth (*Mammuthus primigenius*) and woolly rhinoceros (*Coelodonta antiquitatis*) (Mol et al., 2006, 2008, 2013). Radiocarbon dates of bones of these and other animals raise questions concerning the issues discussed above, such as backgrounds, contamination, age limit and context. As such, the North Sea bones (mostly mammoth) form a unique case study to shed some light on these issues.

Most fossils are collected during fishing expeditions (e.g., Mol and Post, 2010). A difficult issue has always been the context of the finds. Only recently could finds from the Europeul region of the North Sea be studied in a stratigraphic framework (Hijma et al., 2012).

This framework raises questions on ^{14}C dates available for selected fossils. It would mean that all Late Pleistocene terrestrial mammals dating older than around 30,000 years must have been redeposited from their original location. Late Pleistocene marine mammals must be 60–85,000 years old; the available ^{14}C dates, however, are much younger (around 35,000 BP). In addition, all dates from mammoth fossils from the Europeul (the route for large ships entering the harbour of Rotterdam) and adjacent area of the North Sea are older than ca. 34,000 BP (Mol et al., 2008). According

to Hijma et al. (2012), that would mean that these fossils must have been transported. A study by Rijdsdijk et al. (2013) also considers ^{14}C dates anomalously young, based on marine taxa dates obtained by racemization.

This fits in other discussions on “too young” ^{14}C dates and their validity: shells. The datable fraction for these samples is their carbonate. Fossil corals and shells can recrystallize, enabling exchange of carbon from different sources/origin, including ^{14}C . Foraminifera of Eemian age can produce dates significantly younger than 50,000 BP. This open system behaviour appears to be species dependent (Nadeau et al., 2001).

This was already known in the early days of Radiocarbon. Olsson (1989) noted that infinitely old shells date 33,700 BP, corresponding to 1.5% contamination with modern Carbon. Most contamination remains in the outer part of the shells.

In addition, note that also for shell dating the proper background, for ^{14}C dating (infinite age material) must be considered. Using anthracite easily yields similar interpretation problems as for fossil bone collagen discussed above.

This discussion concerning North Sea shell dates does not mean that obtaining good ^{14}C dates for carbonates is impossible, witness the very existence of the calibration curve Intcal13 (Fig. 1). The fact that the calibration curve exists back to 50,000 years ago alone is proof of that.

The curve is for a significant part based on marine samples, in particular the unique and thoroughly measured dataset from the Cariaco Basin (off the coast of Venezuela). These are pristine planktonic foraminifera samples (Hughen et al., 2004). This means that for these carbonates, the ^{14}C content has been measured without problems as discussed above, even beyond 50,000 BP. Apparently these shells do not show open system behaviour. The Radiocarbon Intcal Committee has established unambiguous criteria for marine sample materials (Reimer et al., 2002).

Fig. 2 also shows 9 dates from *Elephas antiquus* found in the North Sea (Mol et al., 2007, 2012). All dates but one obtained for these samples are at the background level of the ^{14}C method. This is consistent with the assumption that this species became extinct not later than the Eemian (Mol et al., 2007) around 100 ka. However, there is one exception yielding a clearly finite ^{14}C date of 37.4 ka BP (Mol et al., 2007). It is true that this is one single measurement, so that it needs an independent confirmation. This was a sample with good quality parameters.

An illustrative example for statistical deliberations is the one but youngest *Elephas* date. In Fig. 2 this date is indicated with an arrow. The date is calculated as 48,200 (+1030, –920) BP. Statistically, this means that there is a 68% chance that the date falls within the range 47,280–49,030 BP (1-sigma criterion); a 95% chance that the date falls within the range 46,360–50,260 BP (2-sigma criterion); and the probability that the true date is infinite (older than 50,260 in this case) is 5%.

This measurement of 48.2 ka BP satisfies the 2-sigma criterion (based on the measured activity and the uncertainty in this value). It yields a finite date, nevertheless there is the possibility that the true age is infinite.

Another example of such a date (taken from Cook et al., 2012) is a ^{14}C date of 49,900 (+5270, –3240) BP. One could interpret this as a published finite bone date of 49.9 ka BP, easily leading to confusion as it might as well represent infinite age. This particular bone is known to date from stage MIS-7, i.e. infinite on the ^{14}C scale. What would one say if the context was not known; infinite age or 49,900 BP? This is the case for the North Sea *Elephas* of 48.8 ka BP: there is no context.

For the users of ^{14}C dates a proper handling of such “borderline dates” is imperative. Considering all aspects discussed here (in particular the proper measurement blank of

bone collagen) it seems best in our opinion to apply an upper limit of 45 ka BP for ^{14}C dates of bones. The Groningen laboratory has adapted this practice, as a pragmatic attempt to diminish further contamination of the literature. When publishing finite dates older than 45 ka BP, one has to show that these are truly finite.

To make progress from this conservative approach, more research is needed. One aspect is cross-checking of bone dates and background samples. For the Sixth International Radiocarbon Intercomparison (SIRI), a series of North Sea bones (mostly woolly mammoth) was collected and analysed. One woolly mammoth femur (a single piece of 8.3 kg) showed a ^{14}C date of 39,860 (+350, –310) BP and had excellent quality parameters.

Anthracite was used as a background to obtain this date. When using a stage 7 bone background, the result becomes 40,490 (+450, –390) BP, which means there is no systematic difference within error. This bone was selected and submitted to the Glasgow Radiocarbon laboratory, which coordinates the intercomparison. Henceforth it became known as “SIRI sample B”. In Groningen the sample B was analysed in duplicate, yielding the dates 39,820 (+350, –310) and 39,520 (+340, –300) BP. These results are well consistent within error, and can be averaged to 39,630 BP. Results from other laboratories are expected (SIRI is presently, 2014, taking place).

Illustrative from a contamination point of view is a mammoth from the North Sea, yielding an exceptional young date: the Preboreal. It was first believed to be sensational, but upon close inspection this sample was contaminated with preservative glue. Glue is a collagen based compound, and as the datable fraction is also collagen, sample and contaminant can not be separated. Samples from the not contaminated, inner parts of the tooth yielded Glacial ^{14}C dates (den Ouden et al., this conference).

The North Sea brings together many aspects of the subject of this paper: validity of ^{14}C dates, degradation, open system behaviour, background questions, different behaviour of different sample materials, calibration, unknown geophysical aspects, and (the lack of) context. Further interdisciplinary research no doubt will increase our understanding of this important heritage “site”.

5. Conclusions

The Radiocarbon dating method has developed during the last 60 years into a reliable dating method for organic sample materials. Recently, the method was extended to the complete dating range of 50,000 years by establishment of the calibration curve known as Intcal13.

The datable sample material includes fossil bone. More than any other material, this category is the subject of discussions concerning validity of the dates, in particular for the older part of the ^{14}C timescale. For paleontology, dealing mostly with pre-Holocene bones, both age range and contamination can easily result in problematic dates; methodological problems become worse for dates closer to the dating limit, as well as for degraded sample materials.

A main issue is the use of proper background materials for collagen, the datable fraction for bone samples. Anthracite is mostly used as a blank. This is not always justified. However, infinitely old bone is not always the correct blank either, considering the occurrence of good and bad quality collagen. Background samples are representative for laboratory treatments, but are not representative for possible carbon exchange (and thus contamination) during degradation or other processes in deposits. In practice, we find that fossil bones with reported ages older than 45,000 BP must be considered with great care, depending on matters such as background material used for the measurement, sample integrity,

context and the non-zero statistical probability the date is infinite on the ^{14}C timescale.

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