The Mesolithic–Neolithic Transition in the Iron Gates, Southeast Europe

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Since its development in 1949, radiocarbon dating has increasingly been used in prehistoric research in order to get a better grip on the chronology of sites, cultures and environmental changes. Refinement of the dating, sampling and calibration methods has continuously created new and challenging perspectives for absolute dating.

In these proceedings the focus lies on the contribution of 14C dates in current Mesolithic research in North-West Europe. Altogether 40 papers dealing with radiocarbon dates from 15 different countries are presented. Major themes are the typo-technological evolution of lithic and bone industries, changes in settlement patterns, burial practices, demography and subsistence, human impact on the Mesolithic environment and the neolithisation process. Some papers also deal with more methodological aspects of 14C dating (e.g. calculation of various reservoir effects, use of cumulative calibrated probability distributions), and related techniques (e.g. stable isotope analysis for palaeodiet reconstruction).

Philippe Crombé, Joris Sergeant and Machteld Bats belong to the Archaeology department of Ghent University. They have all been involved for many years in Final Palaeolithic and Mesolithic research, including field-work (survey and large-scale salvage excavations), and laboratory work (techno-typological analysis, GIS, spatial analysis, etc), mainly in the sandy lowlands and wetlands of NW Belgium, but occasionally also in the Netherlands.

Mark Van Strydonck and Mathieu Boudin belong to the radiocarbon laboratory of the Royal Institute for Cultural Heritage in Brussels. Radiocarbon dating for archaeology and the history of the arts is the main objective of this laboratory, founded in the 1960s. One of the main fields of research in this laboratory is the study of the relationship between the sample and the event (sample selection, integrity and quality on the one hand, event analysis on the other hand).

There exists a long established and close collaboration between the two institutes, which has resulted in joint research projects on dating of Mesolithic pottery food-crusts, antler matlocks, and cremated bones.
CHAPTER TWENTY-FIVE

THE MESOLITHIC–NEOLITHIC TRANSITION IN THE IRON GATES, SOUTHEAST EUROPE: CALIBRATION AND DIETARY ISSUES

GORDON COOK, CLIVE BONSALL, CATRIONA PICKARD, KATHLEEN MCSWEENEY, LÁSZLÓ BARTOSIEWICZ, ADINA BORONEANȚ

Abstract

This paper discusses an aquatic reservoir effect present in Mesolithic human bone samples from the Iron Gates section of the River Danube. Its magnitude has been calculated from a comparison of the $^{14}$C ages of human bones and terrestrial mammal bones from Schela Cladovei, equivalent to 545±70 years for a 100% aquatic diet. From this, using the $\delta^{15}$N value of human bone collagen to estimate the proportion of aquatic food in diet, a correction factor can be applied to the human bone $^{14}$C ages. Reservoir correction makes the resultant $^{14}$C age less precise but more accurate. The reservoir effect is derived from the inclusion of aquatic resources from the River Danube in the diets of the Mesolithic inhabitants. On the basis that the Black Sea became marine around 7400 cal BC, the possibility that part of the reservoir effect derives from anadromous fish species cannot be discounted. Human remains are abundant in the Iron Gates sites and therefore potentially important for construction of archaeological chronologies. Our ability to correct for the aquatic reservoir effect has important implications for establishing accurate chronologies, especially at the Mesolithic–Neolithic transition, which was marked by a significant change in diet and subsistence.
Résumé

Cet article discute d'un effet de réservoir d'origine aquatique dans les échantillons d'os humains mésolithiques provenant de la zone des Portes de Fer sur le Danube. L'intensité de cet effet a été calculée à partir d’une comparaison des âges $^{14}$C d'os humains et de mammifères terrestres provenant de Schela Cladovei, datés de $545\pm 70$ ans avec une diététique à 100% aquatique. À partir de cela, un facteur de correction peut être appliqué aux âges $^{14}$C des os humains en utilisant la valeur $\delta^{15}$N du collagène osseux humain pour estimer la proportion de nourriture d'origine aquatique dans la diète. Cette correction rend les âges $^{14}$C moins précis mais plus exacts. L'effet réservoir dérive de l'inclusion des ressources aquatiques provenant du Danube dans la diète des habitants du Mésolithique. Compte tenu du fait que la Mer Noire est devenue un environnement marin autour de 7400 cal BC, la possibilité qu’une partie de cet effet réservoir dérive de poissons anadromes ne peut pas être écartée. Les restes humains sont abondants dans les sites des Portes de Fer et pour cela leur potentiel sera primordial dans toute construction chronologique archéologique. La possibilité de correction de l’effet de réservoir d’origine aquatique a des implications importantes pour l’établissement de chronologies exactes, et plus particulièrement pour la transition Mésolithique–Néolithique qui a été marquée par des changements cruciaux dans la diététique et dans la subsistance.

Keywords: Iron Gates, Mesolithic, Neolithic, transition, calibration, palaeodiet, Southeast Europe
Mots-clés: Portes de Fer, Mésolithique, Néolithique, transition, calibration, régime alimentaire, sud-est de l’Europe

1. Introduction

The area known as the Iron Gates is the 230km-long section of the Danube Valley forming the border between Romania and Serbia, where dam construction has not only affected river hydrology but also the pattern of archaeological discovery. The region falls naturally into two physiographic zones: 1) The Iron Gates gorge, a 130km-long system of gorges where the Danube breaks through the Carpathian Mountains, extending from the Hungarian Plain to the Iron Gates I dam, and 2) the area downstream between Iron Gates I and II dams where the river is flanked by a broad alluvial plain. Within Southeast Europe the Iron Gates has an unparalleled record of Mesolithic and Early Neolithic settlement, with more than
30 open-air and cave sites belonging to the period from the Late Glacial to the Mid-Holocene (c. 12,000–5500 cal BC), and it is arguably the only area of Southeast Europe where the Mesolithic–Neolithic transition can be studied in detail.

Intensive research since the mid-1960s has provoked considerable debate on a range of issues, particularly those of subsistence patterns and chronology. The main chronological issues are: 1) the history of occupation of the open-air sites (internal chronology) and 2) the timing of the transition from foraging to farming and the dietary and other trends associated with the transition. For several decades, stratigraphy and radiometric $^{14}$C ages made on bulk charcoal samples formed the basis for dating the occupational sequences of individual sites and constructing a regional chronological framework for the Mesolithic and Early Neolithic. However, good series of radiometric $^{14}$C ages were obtained for only a few sites and almost invariably conflicted with the stratigraphic interpretations of the excavators.

One of the most detailed radiometric $^{14}$C sequences is from Lepenski Vir, where charcoal samples from 14 of the trapezoidal-plan, plaster-floored buildings that are unique to this site, gave $^{14}$C ages ranging from 7430±160 to 6560±100 BP. According to Quitta (1972), the charcoal samples were recovered from the “house floors or from the occupation layers immediately above them. Only in a few cases were they recognisable as elements of a house: parts of a burnt beam, for instance, from houses 36 and 37” (Quitta 1972, 205). However, Borić (2002: appendix 1) has provided information based on the original excavation records which suggests that in at least 10 cases (considering only ages with errors of ±100 yr, or less) the charcoal came from contexts contemporaneous with the construction or use of the building. On the basis of the data presented by Borić (2002: appendix 1), the ‘houses’ were built, or at least in use over a period from at least 7335±70 BP (weighted mean of two determinations for ‘House 36’) to 6620±100 BP (single date for ‘House 51’). This interpretation is complicated by two potential sources of error, (i) the charcoal from the ‘houses’ were from long-lived species (oak, Quercus sp., and elm, Ulmus sp.) and could produce an ‘old wood effect’, and (ii) some samples representing structural timbers could have been recycled from earlier buildings. Both of these would result in $^{14}$C ages that are older that the true construction ages of the buildings. It should also be borne in mind that only a relatively small number of contexts were dated and, therefore, may not be representative of the entire time-range over which construction of the trapezoidal buildings took place. An initial series of AMS dates on human bones from burials that supposedly belonged to a later phase than the ‘houses’ (Burials 31a, 32, 35, 44, and 88) gave $^{14}$C ages ranging from 7770±90 to 6910±90 BP. These results implied that either the burials were not later than the ‘houses’ or that the human bones were
influenced by some sort of reservoir effect (Bonsall et al. 1997).

2. Investigating the reservoir effect

The most fundamental assumption in $^{14}$C dating theory is that there is global uniformity in the $^{14}$C-specific activity of all living organisms. The short residence time and rapid mixing of carbon in the atmosphere together with its rapid transfer to the biosphere mean that there is a negligible offset in $^{14}$C activity between the contemporaneous atmosphere and terrestrial biosphere. Therefore, the assumption is valid for the well-mixed atmosphere and the terrestrial flora and fauna that atmospheric carbon supports, provided proper corrections are made for any isotopic fractionation that takes place during $^{14}$C transfer through the food chain. In contrast, the oceans and the plant and animal life that they support represent a rather heterogeneous environment (irrespective of fractionation corrections) that is depleted in $^{14}$C relative to terrestrial flora and fauna. The depletion is brought about by the fact that a body of water can only take up atmospheric carbon while at the surface. Much of the formation of the ocean’s deep waters occurs in the Polar Regions where gaseous exchange and cooling occur. Consequently, there is an increase in density that causes this water to sink away from the surface, thereby removing it from its $^{14}$C source. The slow mixing (thermohaline circulation) of deep-water masses that differ in the time since they were last at the ocean surface, and subsequent upwelling, result in an overall $^{14}$C deficiency within the surface waters of the oceans relative to the atmosphere. The global average surface water depletion is estimated at c. 400 years, but this is known to vary both spatially and temporally (Stuiver and Braziunas, 1993; Austin et al. 1995; Heier-Nielsen et al. 1995; Ascough et al. 2004). The reservoir effects in freshwater bodies can derive from various sources, including dissolution of geological carbonates in the catchment, often termed the ‘hard water effect’ (Shotton 1972) and incorporation of CO$_2$ resulting from volcanic activity in the surrounding environment e.g. Lake Myvatn in Iceland (Ascough et al. 2007).

Any reservoir effect in human bone collagen resulting from consumption of resources from the River Danube could be either marine- (from the Black Sea) or freshwater-related, or indeed a combination of both effects. It should be borne in mind that prior to the establishment of a connection with the Mediterranean, the Black Sea was an enclosed ‘freshwater’ system whose reservoir offset was probably largely derived from the hard water effect. Kwiecien et al. (2006) estimated this effect to be c. 1450 years for the Late Glacial. After marine incursion it has been estimated to be similar to the current global average surface water marine reservoir age of c. 400 years (Kwiecien et al. 2006; Higham et al. 2007). The critical issue here is when the initial marine incursion occurred. Ryan et
al. (1997) initially estimated this to be ca. 7150 BP before applying a reservoir correction of 460 years (c. 5600 cal BC reservoir corrected). However, they later amended this to c. 8400 BP (c. 7500 cal BC) (Major et al. 2006). The further question of whether this incursion constituted a catastrophic flooding appears open to considerable debate (Martin, Leorri and McLaughlin 2007). Thus, human remains dating prior to 8400 BP (reservoir corrected), i.e. ‘Early’ Mesolithic, could have a greater reservoir age than those dated to post-8400 BP if Black Sea fish were an important constituent of the Mesolithic diet. The marine reservoir effect in the Danube would derive from consumption of anadromous fish (e.g. sturgeon spp.) migrating from the Black Sea, and indeed there is abundant evidence of sturgeon fishing in the Iron Gates during most archaeological periods back to the Late Glacial (Nalbant 1970; Clason 1980; Bartosiewicz et al. 1995, 2001; Păunescu 2000; Bartosiewicz and Bonsall 2004; Bartosiewicz, Bonsall and Șișu 2008; Borić and Dimitrijević 2005). The freshwater reservoir effect would derive mainly from the consumption of freshwater fish and molluscs (carp, catfish and freshwater mussels) for which there is also extensive evidence in the archaeological record of the Iron Gates (e.g. Bökönyi 1972, 1978; Clason 1980; Bartosiewicz et al. 1995, 2001).

In Europe, most plants follow the $C_3$ photosynthetic pathway. Therefore, a human diet based on purely terrestrial resources within this region would typically result in bone collagen stable isotope signatures within the ranges of -20.0 to -21.5‰ for $\delta^{13}C$ and +7.0 to +11.0‰ for $\delta^{15}N$, according to the balance of plant versus animal food in the diet (see Bonsall et al., this volume). Stable isotope analyses of human remains from Mesolithic (c. 9600–6000 cal BC) contexts in the Iron Gates are illustrated in table 25-1.

The Mesolithic stable isotope values are significantly heavier than would be expected from a terrestrial diet, even one that was dominated by animal products. It was suggested by Bonsall et al. (1997, 2000) that the diets must have included a substantial aquatic component, either consumed directly in the form of a combination of fish, shellfish, waterfowl and aquatic plants, or indirectly through eating the flesh of dogs (or other terrestrial omnivores/carnivores) that had regularly fed on fish. This interpretation was based on (i) (limited) local food web isotopic studies, (ii) the stable isotope values for modern freshwater fish, and (iii) osteoarchaeological evidence, notably the abundance of fish remains in some of the Iron Gates sites. Borić et al. (2004) published further data on the Mesolithic food web in the Iron Gates, and from this suggested that consumption of some terrestrial animal species such as carnivores (wolf, pine marten and lynx) and suckling omnivores (wild pigs), which have been shown to have bone collagen $\delta^{15}N$ values
Table 25-1: Stable isotope values for AMS $^{14}$C dated archaeological human remains from the Iron Gates.

The Mesolithic data also show a trend toward heavier values in both $\delta^{13}$C and $\delta^{15}$N as the Final Mesolithic is approached. This trend could be interpreted in a number of ways including an increase in the consumption of aquatic resources, a change in the aquatic species targeted, or changes in food preparation techniques. However, little research has been done on the effects of cooking or long-term storage on stable isotope values. Currently, there are insufficient stable isotope data for archaeological fish bones from the Iron Gates to offer definitive conclusions about the relative contributions of the resources that were consumed, e.g. particular species or groups of fish (anadromous versus freshwater). The few data that are available would tend to indicate that sturgeon made a significant contribution to diet and, as stated above, there is evidence of sturgeon fishing at a number of Stone Age (Late Glacial to Mid-Holocene) sites within the Iron Gates.

The existence of a reservoir effect in the Mesolithic human bone collagen would argue against the inclusion of large amounts of meat from terrestrial
carnivores and suckling omnivores in the diet since this would not generate an age offset. Therefore, realistically, such an effect could only be derived from the consumption of aquatic resources.

To establish the existence of a reservoir effect requires comparison of contemporaneous terrestrial and aquatic material. While there are abundant fish and mammalian bones at some of the Iron Gates sites, it is only very rarely that contemporaneity between the two can be demonstrated. However, the Romanian–British excavations at Schela Cladovei (Romania) provided ideal samples for this purpose (Bonsall et al. 1997). These comprised the burials of individuals that had died from arrow injuries. The arrowheads were found embedded in bones or in positions suggesting they had penetrated vital organs. These arrowheads were made from bones of terrestrial ungulates (deer, wild boar) which allow comparison of human bone (with reservoir effect) and mammalian bone (without reservoir effect). Figure 25-1 illustrates the ages of the five pairs of samples that were analysed. There were two arrowheads in the human remains constituting Pairs 4 and 5.

These results clearly demonstrate that there is a reservoir effect and this could not have derived from consumption of terrestrial resources. Figure 25-2, which plots the age offset between directly-dated Mesolithic human bones and animal bone tools from Schela Cladovei, also clearly shows an offset.

3. Estimating the reservoir effect

To estimate the reservoir effect, it is necessary to assume certain dietary end-members. For a 100% terrestrial, mixed farming diet, the stable isotope end-member values that were chosen comprised: \( \delta^{15}N = +8.3‰ \) and \( \delta^{13}C = -20.4‰ \). These values are based on the analysis of individuals, all dating to approximately 5400 BP, from Ajdovska Jama (Pagan’s Cave), southeast Slovenia (Bonsall et al. 2007). These values differ slightly from those published in Cook et al. (2001). The 100% aquatic diet stable isotope end-members are more open to question. The values chosen were \( \delta^{15}N = +17.0‰ \) and \( \delta^{13}C = -17.5‰ \). These are the heaviest values measured in any of our studies within the Iron Gates. Carbon atoms in human bone collagen derive primarily from the protein component in the diet when this component is adequate. However, in low protein diets, a significant proportion of the carbon atoms will derive from carbohydrates and lipids (Ambrose and Norr 1993; Bonsall et al., this volume). In contrast, nitrogen atoms in human bone collagen are all derived from the protein component of the diet; therefore the relationship between a 100% terrestrial and a 100% aquatic diet should be close to linear for \( \delta^{15}N \). Nevertheless, figure 25-3 illustrates a relatively good linear
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relationship between δ^{13}C and δ^{15}N, perhaps further confirmation that the Mesolithic diet within the Iron Gates was high in protein (cf. Bonsall et al. 1997).

Within the five pairs of data illustrated in figure 25-1, there are three human bone samples with δ^{15}N values close to +15‰ (average of +15.1‰). On a linear scale between the terrestrial and aquatic end-member values of +8.3 and +17.0‰, this equates to a 78% aquatic diet. The average age offset between the three human bones and the associated ungulate bone points is 425±55 years, which therefore equates to a 78% aquatic diet. By extension, a 100% aquatic diet corresponds to 545±70 years. It follows that any radiocarbon age for human bone collagen can be corrected for the reservoir effect provided the δ^{15}N value is known.

4. Application of the reservoir correction

Using the linear relationship described above we corrected (using Method 1 of Cook et al. 2002) all of the radiocarbon dated human remains from Lepenski Vir for which we had δ^{15}N values for the human bone collagen. These results are presented in table 25-2 in non-reservoir-corrected chronological sequence. Table 25-3 presents the reservoir-corrected results in chronological order.

The corrected 14C ages follow a continuous series with no apparent gap between the Final Mesolithic and the Early Neolithic, which is exactly what would be expected if sampling across a transition. The stable isotope data (with three exceptions) also form a logical sequence with the heavy values paired to the earliest ages and the lighter ones with the younger ages. The two individuals with intermediate δ^{15}N values (ages at death were >40 years) could be incoming Early Neolithic farmers whose collagen had partially turned over to represent the aquatic diet, or alternatively, they could have been incoming Mesolithic hunter-gatherers, still with aquatic diet, but perhaps consuming less fish, different fish species, or different sized fish. The third individual with the lightest stable isotope values died at a younger age and, therefore, there would have been less time for the stable isotope signatures in the bone collagen to change. It should be noted that these three individuals were all buried in the characteristic Late Mesolithic manner of extended, supine inhumation. An alternative explanation could be that the lifetimes of these three individuals overlapped with ‘false starts’ in agriculture in the locality.
Fig. 25-1: Age offset between paired human bone (●) and ungulate bone points (+).
Fig. 25-2: Age offset between directly dated Mesolithic human bones (●) and animal bone tools (+) from Schela Cladovei, Romania.
Fig. 25-3: Relationship between $\delta^{13}$C and $\delta^{15}$N for Late Mesolithic and Early Neolithic human bone collagen samples from Lepenski Vir (see Table 25-2).
Fig. 25-4: Linear function of percentage aquatic diet versus δ¹⁵N in human bone collagen.
The Mesolithic-Neolithic Transition in the Iron Gates: Calibration and Dietary Issues

<table>
<thead>
<tr>
<th>Uncorrected Age (years BP±1σ)</th>
<th>δ¹³C (‰)</th>
<th>δ¹⁵N (‰)</th>
<th>Aquatic Diet (%)</th>
<th>Corrected Age (years BP±1σ)</th>
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Table 25-2: Correction of Lepenski Vir Late Mesolithic and Early Neolithic human bone Ages for Reservoir Effect (RE).

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<thead>
<tr>
<th>Corrected Age (years BP±1σ)</th>
<th>δ¹⁵N (‰)</th>
<th>δ¹³C (‰)</th>
<th>Corrected Age (years BP±1σ)</th>
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<th>δ¹³C (‰)</th>
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Table 25-3: Corrected Lepenski Vir Late Mesolithic and Early Neolithic human bone ages and stable isotope values in chronological order.
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5. Is the magnitude of the reservoir correction realistic?

The range of $^{14}$C ages for the charcoal samples from Lepenski Vir was 7430±160 to 6560±100 BP while the ages for the human bones that purported to come from a later phase ranged from 7770±90 to 6910±90 BP. When the reservoir correction was applied to the human bone ages, this shifted the range to 7375±80 to 6730±95 BP. This age range and the fact that some of the Lepenski Vir III burials had ‘Mesolithic’ dietary signals raises questions about the phasing of the burials. It also raises doubts about the stratigraphic integrity of Lepenski Vir III, because the ages are no different from phases I and II.

When the reservoir correction is applied to the human bone ages from the Schela Cladovei human bone/ungulate bone point pairs, this produces age pairs that are indistinguishable. It could be argued that there is some circularity in this argument since the differences in age between some of the pairs was used in the calculation of the diet-based reservoir correction; however, if the linear function in $\delta^{15}$N that was assumed did not hold true, then the calculation of the 100% dietary offset and its application to the human/ungulate bone pairs would not necessarily result in pairs that did not differ statistically in age. Furthermore, subsequent application of the correction to all the human bone ages from Schela Cladovei and their comparison with the $^{14}$C ages of all directly-dated terrestrial animal bone artefacts from the site demonstrates a much closer agreement in ages (Fig. 25-5) than prior to the correction (Fig. 25-2).

<table>
<thead>
<tr>
<th>Pair Number</th>
<th>Uncorrected human bone $^{14}$C ages (Years BP±1σ)</th>
<th>Reservoir corrected human bone $^{14}$C ages (Years BP±1σ)</th>
<th>Ungulate bone $^{14}$C ages (Years BP±1σ)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>7790±100</td>
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<td>8380±80</td>
<td>7960±110</td>
<td>7915±65, 7780±75</td>
</tr>
<tr>
<td>5</td>
<td>8200±70</td>
<td>7760±100</td>
<td>7905±60, 7805±70</td>
</tr>
</tbody>
</table>

Table 25-4: Comparison of uncorrected and corrected human bone $^{14}$C ages with $^{14}$C ages for associated ungulate bone projectile points from Schela Cladovei, Romania.
6. Conclusions

Calculation of a reservoir effect from the comparison of the $^{14}$C ages of human bones and terrestrial mammal bones from Late Mesolithic contexts at Schela Cladovei in the Iron Gates yields a value of 545±70 years. Application of the reservoir correction makes the resultant $^{14}$C age less precise (i.e. larger error term) but more accurate (mean age closer to the true value). This effect is derived from the inclusion of aquatic resources (primarily fish) from the River Danube in the diets of the Mesolithic inhabitants of the Iron Gates. On the basis that the Black Sea became marine around 8400 BP (c. 7400 cal BC) the possibility that at least...
part of the reservoir effect derives from anadromous fish species, such as sturgeons, cannot be discounted. In fact, given the large size of these fish and the ethnohistorical evidence for their presence in large numbers in the Iron Gates reach of the Danube, it is likely that sturgeons did contribute significantly to this effect. This is a modification of the arguments presented by Cook et al. (2001, 2002) that the reservoir effect was exclusively freshwater in origin. At that time the popular view was that a giant freshwater lake had occupied the Black Sea basin up until c. 6700 BP (Ryan et al. 1997). Subsequent work suggests that the conversion of the Black Sea into a marine system occurred much earlier, at around 8400 BP (Major et al. 2006) or before (Bahr et al. 2006).

An argument has also been presented for multiple marine incursions into the Black Sea from the Mediterranean related to freshwater discharge events during the Holocene (Martin, Leorri and McLaughlin 2007). Such fluctuating salinity would influence the food sources available to Black Sea sturgeon and, conceivably, the stable isotope values of their tissues (including bone). This has implications for calculating the size of the reservoir effect over time. It is interesting to note that our data for the Iron Gates show a significant enrichment in both $\delta^{15}$N and $\delta^{13}$C values of human bone collagen between the Early and Late Mesolithic (Table 25-1) which corresponds approximately to the timing of the initial Holocene marine incursion, and may therefore reflect this.

We discount the alternative theory for the high $\delta^{15}$N values being derived from consumption of certain carnivores and suckling omnivores, on the basis that this would not generate the very obvious age offsets that exist between human bone ages and those of generally contemporaneous terrestrial animal bones and wood charcoal.

More generally, these results have implications for absolute chronologies that are based on the dating of human remains, where those remains derive from a population or populations whose members consumed significant aquatic resources. Such a reservoir effect will not be confined to the Mesolithic, but could apply to any period where there was a reliance on riverine and/or lacustrine resources. It also needs to be stressed that aquatic protein intake can be direct, or indirect through consumption of the flesh of scavengers such as dogs and pigs.

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