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Problems of dating human bones from the Iron Gates

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It is widely recognized that when marine resources form a significant proportion of the human diet, this results in radiocarbon ages for human remains that are significantly older than the contemporary atmosphere. While there has been widespread assessment of marine ¹⁴C reservoir ages, there has been little study of the freshwater equivalent. However, recent analyses of human bone from archaeological sites in the Danube Valley have confirmed the existence of a large freshwater ¹⁴C reservoir effect.

Key-words: radiocarbon dating, reservoir effect, human bones, Iron Gates, diet, stable isotopes, Mesolithic, Lepenski Vir, Schela Cladovei, Vlasac

In studies of the Iron Gates Stone Age (FIGURE 1) there is considerable conflict between the archaeological phasing of sites and radiocarbon dating-based chronologies derived from measurements made on charcoal and human bone samples, For example, at Lepenski Vir, Srejović (1972) identified five occupation phases: Proto-Lepenski Vir, Lepenski Vir I, II, IIIa and IIIb, Phases I and II were assigned to the Mesolithic, while phases IIIa and IIIb, by virtue of the presence of both pottery and bones of domesticated animals, were assigned to the Early Neolithic, However, a series of charcoal samples from contexts associated with houses from the Mesolithic (phases I and II) produced ¹⁴C ages between 6560 and 7360 BP (Quitta 1972), similar to those for Early Neolithic (Starčevo-Körös-Criş) sites in the surrounding regions. Srejović (1972; 1989) rejected these ¹⁴C ages as approximately 500 years too young, while other researchers (Voytek & Tringham 1989; Chapman 1992; Radovanović 1996) have accepted them and interpret Lepenski Vir I and II as representing the latest phases of the Mesolithic in the Iron Gates region. Their view is that the gorge was rich in natural resources but not immediately attractive to farming, and therefore continued to be occupied by huntergatherer communities. A further hypothesis was proposed by Milisauskas (1978), namely that the structures comprising Lepenski Vir I and II were the remains of houses built by a sedentary farming community that had dug the foundations into earlier hunter-gatherer deposits, thereby producing a mix of materials from Mesolithic and Early Neolithic occupations. Subsequent AMS dating of human remains assigned to the later phase III of Lepenski Vir produced ages between 6910 and 7770 BP which puts them out of sequence with the charcoal samples assigned to phases I and II (Bonsall et al. 1997). At Vlasac, three phases of Mesolithic occupation (Vlasac I-III) and traces of Early Neolithic settlement (Vlasac IV) were designated (Srejović & Letica 1978; Prinz 1987). At this site, a series of 15 ¹⁴C age measurements made on charcoal samples assigned to phases I-III produced an age range of 6790±100 BP to 7935±60 BP, while human bone samples from phases I and III produced ages between 8000±100 BP and 10,240±120 BP (Bonsall et al. 1997). At Schela Cladovei, in areas excavated by Boroneant, evidence of two Mesolithic phases (Schela Cladovei I and II) and at least two phases of Early Neolithic settlement were recognised (Boroneant 1970; 1973; 1989; 1990). Here, a similar pattern is observed, although on a much more limited data set. 14C age measurements

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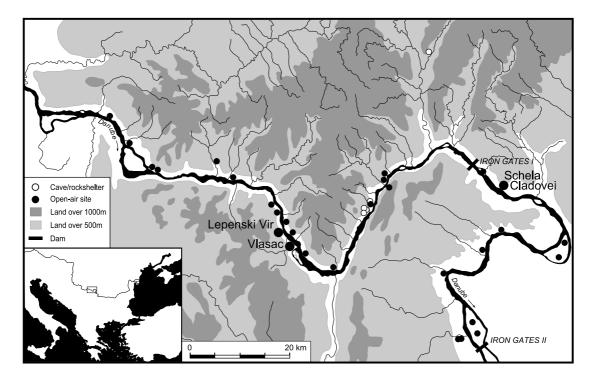


Figure 1. Stone Age sites in the Iron Gates.

on charcoal samples from hearth deposits assigned to the second Mesolithic phase produced ages of 8150±80 BP and 7580±90 BP while a series of measurements made on human bones from the Mesolithic produced an age range of 8290±105 BP to 8570±105 BP (Bonsall *et al.* 1997).

Leaving aside the documented difficulties in attributing burials to different occupational phases, e.g. at Vlasac (Srejović & Letica 1978) or suggestions of incorrect phasing of burials, e.g. at Lepenski Vir (Bonsall et al. 1997), the very obvious trend in these data is that the age range for the human bone samples is always somewhat earlier than the range for the charcoals that were derived from either earlier contexts or contexts that are contemporary with the human bones. Even if the samples were contemporary in context, it is normally expected that either

- i the charcoal samples will produce similar ¹⁴C ages if short-lived species and/or roundwood were specifically selected for measurement, or
- ii they will produce older ages because of the inclusion of long-lived species (old wood effect), or

iii they will produce older ages because of the inclusion of timber that had been used previously for a significant period of time for construction or similar purposes, prior to burning.

Therefore, if the ¹⁴C ages of the charcoal samples are accepted as being correct, then the ages of the human bone samples must be too old which, in turn, suggests the possibility that the human diet may have included material from a reservoir that differed in ¹⁴C specific activity from the contemporary atmosphere.

At all of the above sites, the stable isotope evidence derived from human bone collagen samples indicates that there was a significant non-terrestrial (i.e. aquatic) component to the Mesolithic diet. For samples assigned to the Lepenski Vir Mesolithic, δ^{13} C values range between $-18\cdot1$ and $-20\cdot2\%$ (Bonsall *et al.* 1997). These are slightly enriched compared to what would be expected from a predominantly terrestrial-based diet (Johansen *et al.* 1986). The δ^{15} N values range between $+10\cdot0$ and $+17\cdot0\%$ (Bonsall *et al.* 1997) and many of them are >+14% and are significantly enriched compared to those for a typical terrestrial diet. For exam-

ple, in a study of the dietary habits of Neolithic people living in Slovenia, Ogrinc (1999) obtained an average $\delta^{15}N$ value for adults of approximately +7.5% while Mays (1998) estimated +8 to +10% as the likely δ^{15} N range resulting from a pure C₃ terrestrial diet. At Vlasac, a similar trend was observed in which δ^{13} C values range between -18.2 and -19.5% and $\delta^{15}N$ values range between +13.5 and +15.9% (Bonsall *et al.* 1997). while at Schela Cladovei the δ¹³C values range from -19.2 to -20.0% (Bonsall *et al.* 1997) and the $\delta^{15}N$ values range from +14.9 to +16.0%. All of the above $\delta^{15}N$ data are indicative of a significant dietary component derived from a complex food web in which trophic level increases from primary producer to top carnivore are particularly pronounced. This would be typical of a marine ecosystem (Schoeninger & DeNiro 1984) and, indeed, there is considerable evidence of fish consumption at the Iron Gates sites, which in principle might have included anadromous species (i.e. sturgeon) from the Black Sea. However, this can be discounted for two reasons:

- i the $\delta^{13}C$ values are lower than would be expected for a diet that included significant amounts of marine foodstuffs, and
- ii there is good evidence that the Black Sea was not a marine environment at that time and would not therefore have provided marine reservoir isotopic signatures.

For example, Richards and Hedges (1999) suggest that for a theoretical diet producing a $\delta^{15}N$ value of + 15‰, the $\delta^{13}C$ value should be in the region of approximately –16‰. In addition, from actual measurements made on human bone collagen samples from late Mesolithic sites along the Atlantic coast of Europe, their data indicate that a $\delta^{15}N$ value of +15‰ should be accompanied by a $\delta^{13}C$ value of approximately –15‰.

Lanting & van der Plicht (1998) discuss the likely $\delta^{15}N$ and $\delta^{13}C$ values that would be expected as a result of various diets and these are summarised in Table 1. Their data are consistent with the conclusion of Bonsall *et al.* (1997) that the most likely source of dietary protein is freshwater fish from the Danube. This need not rule out the consumption of migratory sturgeon, if the Black Sea were a freshwater lake at the time. This would be consistent with the evidence of Ryan *et al.* (1997) that the drowning of the Black Sea shelf by seawater

did not take place until around 6700 BP (allowing for reservoir correction — cf. Jones & Gagnon 1994) when the Mediterranean rose to the Bosporus sill. Unfortunately, the existing stable isotope data for fish bone collagen from the Iron Gates are limited to three analyses of samples from Lepenski Vir and these show no consistent pattern, with δ^{13} C values varying between -26.3 and -15.7% and δ^{15} N values varying between +8.2 and +12.9% (Bonsall et al. 1997). Therefore, it is not possible to comment on the source of the fish on the basis of these analyses.

If a +3·4‰ trophic level shift is employed between freshwater fish and human bone collagen (Minagawa & Wada 1984), this certainly indicates that $\delta^{15}N$ values for fish of approximately +11‰, or greater, could result in human bone collagen values of >+14%. While this type of data for fish from the Iron Gates is limited, such values are not uncommon. Iacumin et al. (1998) and Pate (1998) report δ^{15} N values of about +12% for Lake Nasser and South Australia fish, respectively. Doucett et al. (1999) quote a range of +7.4 to +16.8% for brook trout from what they considered to be a mixed anadromous/non-anadromous population, Dufour et al. (1999) report δ^{15} N values >+13‰ for fish from Lake Geneva and Lake Constance. while Hobson & Welch (1995) report δ¹⁵N values for large char collected from a high Arctic lake of >+14%. The enrichment of any species will, of course, depend on the complexity of the food web and its trophic level within the

This reliance by Mesolithic populations on freshwater food sources obviously implies that the riverine reservoir is the one that differs in ¹⁴C specific activity from the contemporary atmosphere and that an age correction may have to be applied to the human bones to take this

diet	δ ¹³ C (‰)	$\delta^{15}N$ (‰)
C ₃ vegetable	-21	+5
flesh of C ₃ herbivores	-18	+8
C ₄ vegetable	- 7	+5
marine food	-13	+18
freshwater fish (river)	-24	+16
freshwater fish (lake)	-20	+16

Table 1. Mean bone collagen values of $\delta^{13}C$ and $\delta^{15}N$ to be expected in 100% diets of the listed categories (from Lanting & van der Plicht 1998).

effect into account, Although marine reservoir effects are well documented (Harkness 1983; Tauber 1983; Arneborg et al. 1999), much less is known about freshwater reservoir effects, These have been reported for the Netherlands by Lanting & van der Plicht (1998) who noted reservoir ages of up to 4430 years in canals and 2000 years approximately in rivers such as the Waal and Maas. The authors also demonstrated significant reservoir effects in the skeletons of the historically dated, 11–13th-century Counts of Holland which they ascribed to the consumption of freshwater fish.

Bonsall *et al.* (1997) have suggested that a freshwater reservoir effect for the Iron Gates Stone Age sites could most easily be tested by comparing age measurements on human bones with those on artefacts of terrestrial (ungulate) animal bone found in the same graves and, indeed, Schela Cladovei provides material ideal for investigating this problem, Cook *et al.* (in press) present ¹⁴C ages for this material, which comprises a suite of human bone samples and associated ungulate bones (Table 2).

The human bones were from single inhumation burials while the ungulate bones were found in direct association with the skeletons. The associations were either bone projectile points embedded in human bone (FIGURE 2) or bone projectile points found immediately adjacent to bones of articulated skeletons (which may originally have been embedded in the soft tissue surrounding the bones). In all cases, the bone points may have been the actual cause of death. In the case of the 'embedded' projectile

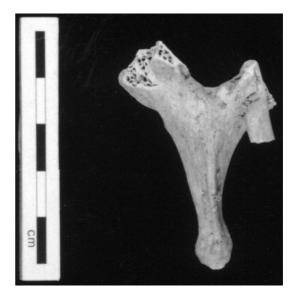


Figure 2. Fragment of a bone projectile point embedded in the lamina of a thoracic vertebra of a Mesolithic human skeleton from Schela Cladovei, Romania.

points, there can be no doubt about the reliability of the association between the human bones and the bone artefacts. In open-air archaeological sites where soil-forming processes have been active there is always a possibility that very small objects can be moved from their original positions, e.g. by the actions of roots or earthworms. The bone points not embedded in human bones are not thought to have been significantly affected by such processes since at Schela Cladovei such artefacts are found al-

human bone samples

- 1.1 bone sample from skeleton, excavated in 1967
- 2.1 bone sample from skeleton, excavated in 1967
- 3.1 bone sample from skeleton, excavated in 1991
- 4.1 bone sample from skeleton, excavated in 1995
- 5.1 bone sample from skeleton, excavated in 1996

associated terrestrial (ungulate) bone samples

- 1.2 bone point **embedded** in thoracic vertebra of skeleton
- 2.2 bone point **embedded** in lumbar vertebra of skeleton
- 3.2 bone point **embedded** in thoracic vertebra of skeleton
- 4.2 bone point **embedded** in left innominate of skeleton
- 4.3 bone point found **adjacent** to skull of skeleton
- 5.2 bone point found adjacent to proximal end of femur of skeleton
- 5.3 bone point found between lowermost vertebra and left innominate of skeleton

Table 2. Samples from Schela Cladovei for ¹⁴C analysis: human bones from single inhumation burials and artefacts made from terrestrial mammal {ungulate} bone found in direct association with the skeletons.

most exclusively with burials and are extremely rare in other contexts, and since the burials themselves appear undisturbed by soil processes. Therefore, there is no reason to doubt any of the associations between the projectile points and human remains, and so ¹⁴C analysis of the human bones and comparison with the ages derived from the directly associated ungulate bones would provide firm evidence of the existence of a dietary induced reservoir age. Results of the ¹⁴C and stable isotope analyses are presented in Table 3.

The human bone collagen δ^{13} C values ranged from $-18\cdot2$ to $-19\cdot5\%$ and are of a similar range to the earlier data of Bonsall *et al.* (1997). The δ^{15} N values are in the range $+13\cdot2$ to $+15\cdot3$ and are, on average, heavier than the ungulate samples by $8\cdot6\%$. Allowing for trophic level shifts, both the δ^{13} C and δ^{15} N values for the human bone collagen are consistent with a diet whose protein is dominated by freshwater fish.

The ¹⁴C results indicate that the human bone collagen samples are always significantly older than the ungulate samples by approximately 300–500 years, clearly demonstrating a freshwater reservoir effect. The weighted mean offset between the paired samples is 440±45 years and subtraction of this reservoir age will bring the age of the human bone samples closer to the true ¹⁴C age of the contemporary terrestrial biota. However, this necessarily assumes that individuals in the Mesolithic population all had the same proportion of aquatic protein in their

diet. A more elegant approach would be to derive a relationship between each individual's level of aquatic diet and the magnitude of the age offset. Ambrose & Norr (1993) have concluded that in low-protein diets the $\delta^{13}C$ in collagen may not represent only the sources of protein in the diet, because of carbon contributions from carbohydrates and lipids. This means that a linear mixing model between $\delta^{13}C$ in bone collagen of the consumer and in the different components of the diet cannot be used in palaeodietary studies.

However, this problem should not apply to nitrogen as a higher animal's principal source of nitrogen is the protein that it consumes in its diet (Conn & Stumpf 1972). Cook et al. (in press) derived a simple linear relationship between δ^{15} N and age offset. The basic premise is that the more aquatic food intake an individual has, the greater the $\delta^{15}N$ enrichment in their bone collagen and the greater the aquatic reservoir effect. To accomplish this, an end-point of +8% for 100% terrestrial diet was assumed. This is based on the studies of Ogrinc (1999) and Mays (1998). For 100% aquatic diet, Cook et al. (in press) assumed +17‰, which is the highest $\delta^{15}N$ value measured in an adult from the Iron Gates region (Bonsall et al. 1997, and unpublished results).

Of the five human bone collagen samples presented in Table 3, three have $\delta^{15}N$ values that are statistically indistinguishable (+15·1, +15·0 and +15·3‰). The average (+15·1‰), based

lab ID	sample ID	bone type	δ ¹³ C (‰)	δ ¹⁵ N (‰)	¹⁴ C age	age offset
OxA-8502	IG(D)1.1	human	-18⋅6	13·2	8300±60	510±117
OxA-8579	IG(D)1.2	ungulate	-20⋅6	5·0	7790±100	
OxA-8547	IG(D)2.1	human	-19⋅3	13⋅9	8240±60	470±247
OxA-8580	IG(D)2.2	ungulate	-20⋅8	6⋅1	7770±240	
OxA-8581	IG(D)3.1	human	-19·5	15·1	8330±75	450±300
OxA-8582	IG(D)3.2	ungulate	-22·0	9·4	7880±290	
OxA-8583 OxA-8584 OxA-8585	IG(D)4.1 IG(D)4.2 IG(D)4.3	human ungulate ungulate	-18.5 -21.5 -20.9	15.0 4.7 4.5	8380±80 7915±65 7780±75	465±103 600±110
OxA-8548 OxA-8549 OxA-8550	IG(D)5.1 IG(D)5.2 IG(D)5.3	human ungulate ungulate	-18.2 -20.4 -21.2	15·3 7·7 4·7	8200±70 7905±60 7805±70	295±92 395±99

Table 3. $\delta^{13}C$ and $\delta^{15}N$ measurements, ^{14}C ages and ^{14}C age offsets in human bone and associated ungulate bone samples from Schela Cladovei. All ^{14}C ages and age offsets are expressed in conventional radiocarbon years BP (before 1950). The errors are expressed at the one sigma level of confidence.

on the linear relationship between +8 and +17‰, corresponds to 79% aquatic diet, In turn, these samples have a weighted mean age offset from the associated ungulate bones of 425±55 ¹⁴C years. Therefore, the reservoir age for 100% aquatic diet would approximate to 540±70 ¹⁴C years (Method 1). This provides the potential to correct individual human bone ages from the Iron Gates, thereby producing age estimates that reflect more closely the terrestrial environment. The foregoing hypothesis is at present based on a very limited data set and could be considered slightly speculative. The alternative strategy discussed above was to use all of the data in Table 3 (440±45 years offset) and to apply this to all the human bone ages. However, it is clear from the spread in the $\delta^{15}N$ data that temporal changes in diet did occur. A compromise between these two approaches would be to assume that any human bone collagen δ^{15} N values >+13% represent 100% aquatic diet and any values between +10 and +13% represent 50% aquatic diet. On the basis of this calculation, 100% aquatic diet would give a reservoir age of 440±45 years and 50% aquatic diet would equate to 220±23 years. These results are presented as Method 2 in TABLES 4 & 5

A third strategy that has also been employed was to take a weighted mean ¹⁴C age for all the bone artefacts from Schela Cladovei relating to the Mesolithic (19 analyses) and to compare these with all the Mesolithic human bone 14C ages (FIGURE 3). It should be noted that all the artefact and human bone samples came from an area of less than 50x50 m. On this occasion, only those human bone samples with $\delta^{15}N$ values of $\geq +15\%$ were considered (10 analyses). The bone artefact ages ranged from 8105±60 BP to 7460±75 BP with a weighted mean of 7878±42 BP while the human bone ages ranged from 8550±105 to 8200±70 BP with a weighted mean of 8401±39 BP. The average $\delta^{15}N$ value was 15·4‰ which corresponds to an 82% aquatic diet which in turn gives a reservoir correction of 638±70 years (Method 3). In this case, the

lab ID	ske	leton	¹⁴ C age	δ ¹⁵ N (‰)	aquatic diet (‰)*	corrected ¹⁴ C age Method 1	corrected ¹⁴ C age Method 2	corrected ¹⁴ C age Method 3
OxA-58	24	72	10240±120	14.5	72	9850±130	9800±130	9800±130
OxA-58	22	51a	8760±110	14.4	71	8380±120	8320±120	8320±120
OxA-58	26	83	8200±90	14.6	73	7810±105	7760±100	7750±105
OxA-58	23	54	8170±100	14.9	77	7750±115	7730±110	7700±115
OxA-58	25	24	8000±90	14.7	74	7600±115	7560±110	7540±115

Table 4. Correcting human bone dates from Vlasac for the aquatic reservoir age using the $\delta^{15}N$ value to determine percentage aquatic diet. All ¹⁴C ages are expressed in conventional radiocarbon years BP (before 1950). The errors are expressed at the one sigma level of confidence.

lab ID sko	eleton	¹⁴ C age	δ ¹³ C (‰)	δ ¹⁵ N (‰)	aquatic diet (‰)*	corrected ¹⁴ C age Method 1	corrected ¹⁴ C age Method 2	corrected ¹⁴ C age Method 3
OxA-5827	31a	7770±90	-18.7	15.7	86	7310±108	7230±101a	7240±108
OxA-5830	44	7590±90	-18.9	15.3	81	7150±106	7150±101a	7090±106
OxA-5828	32	7270±90	-19.5	11.9	43	7040±95	$7050 \pm 93^{\rm b}$	7010±95
OxA-5831	88	7130±90	-20.2	10.9	32	6960±93	6910±93 ^b	6930±93
OxA-5829	35	6910±90	-19.7	11.2	36	6720±93	$6690 \pm 93^{\rm b}$	6690±93

Table 5. Correcting human bone dates from Lepenski Vir III for the aquatic reservoir age using the $\delta^{15}N$ value to determine percentage aquatic diet. All ^{14}C ages are expressed in conventional radiocarbon years BP (before 1950). The errors are expressed at the one sigma level of confidence.

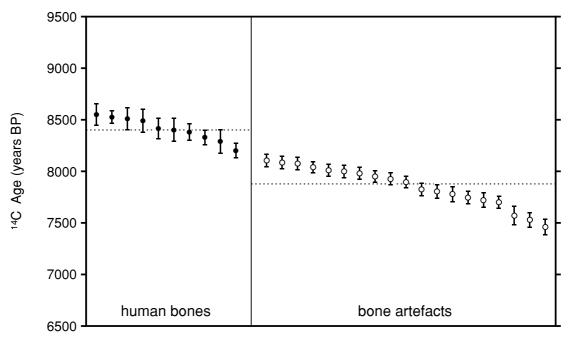


FIGURE 3. Comparison of radiocarbon age ranges of Mesolithic human bones and bone artefacts from Schela Cladovei. Romania. Errors are shown at one standard deviation. The dashed lines represent the weighted means of the radiocarbon ages in each series.

assumption has to be made that the human bones and the artefacts encompass the same length of time within the Mesolithic.

Table 4 illustrates ^{14}C ages for a series of human burials from Vlasac in which each individual has a $\delta^{15}\text{N}$ value that is typical of a high intake of aquatic food. Based on the scale of 8‰ for 100% terrestrial diet and 17‰ for 100% aquatic diet, this indicates between 71 and 77% aquatic diet for the 5 individuals. These estimates of percentage aquatic diet were used in the Methods 1 and 3 calculations. All of the $\delta^{15}\text{N}$ values exceed 13‰ and for Method 2 we therefore applied 100% reservoir correction to all ^{14}C measurements.

Using all three age corrections, the human bone ages now overlap the 'older' series of $^{14}\mathrm{C}$ measurements on charcoal samples from Vlasac which range from 7935 to 7440 BP (see Bonsall et al. 1997). The 'younger' series of charcoal dates from Vlasac (7000–6790 BP) are similar to the mixed aquatic/terrestrial ('Neolithic') corrected ages from Lepenski Vir (see Table 5 below). It is of interest to note that burial 72 which is a minimum of approximately 1500 radiocarbon years older than the other burials has a $\delta^{15}\mathrm{N}$ value that is indistinguishable from

the others in the series. The δ^{13} C value (Bonsall *et al.* 1997) is also typical of the others in this series. This indicates little change in diet over at least a 2000-year period during the Mesolithic.

Table 5 illustrates a series of human bone ages and associated stable isotope values for burials attributed to phase III of Lepenski Vir (Bonsall et al. 1997). These ages span a period during which the population appears to have undergone a significant change in dietary/subsistence patterns (aquatic to mixed aquatic/terrestrial) and can be divided into two groups. Burials 32, 35 and 88 have stable isotope values typical of a mixed diet while burials 31a and 44 are characterized by more enriched $\delta^{13}C$ and δ^{15} N values, typical of a primarily aquatic diet. The latter group is also older than the first group. Calculation of the percentage aquatic diet indicates >80% in samples 31a and 44 and <45% in the other three samples. Leaving aside the question of whether this dietary shift represents the beginning of farming in the gorge, it is interesting to note the effect of correcting the age measurements. Application of the δ^{15} N-derived aquatic reservoir corrections by the three methods discussed above results in the ages quoted in the final three columns of Table 5.

The important aspects to note here are:

- 1 Using any correction method, there is now no significant age difference between the two groups of samples. The corrected ¹⁴C ages form a continuous sequence and this is what would be expected if sampling across a transition (in a continuous series).
- 2 The corrected ages indicate a dietary transition at around 7100 BP.
- 3 The corrected ages, which span the period from 7310 to 6720 BP (or 7330 to 6690 BP or 7240 to 6690 BP) are now in good agreement with the previous radiometric measurements made on charcoal samples from house floors/hearths of Lepenski Vir I and II which spanned 7360 to 6560 BP. However, an important point to note is that many of the charcoal samples were oak which may include 'old timber'. Therefore, the radiocarbon ages of the charcoal samples are conceivably overestimates. Speculatively, a more realistic estimate would be 7200-6400 BP, thereby implying that a significant proportion of the houses were constructed around or after the dietary shift.
- 4 If one accepts that the dietary shift represents a shift from Mesolithic to Neolithic then the data contradict the conventional view (Voytek & Tringham 1989; Chapman 1992; Radovanović 1996) of a population

that did not adopt farming until several hundred years after its introduction into surrounding areas.

More generally, these results demonstrate that it is not only a marine diet that may induce a reservoir age in human collagen but that a freshwater aquatic diet can have a similar and indeed potentially a greater influence. This has important implications for chronologies that are based on dating human remains where the population has a requirement for a significant freshwater component within their diet. This effect will not necessarily be confined to the Mesolithic, but to any period where there was a reliance on freshwater food sources. For example, Lanting & van der Plicht (1998) have suggested that this may be the case for Medieval and later populations in the Netherlands. While the routine measurement of δ^{13} C may help to identify a marine diet (because of the potential for a large shift), it is unlikely to identify a freshwater diet. This reinforces the conclusion of Lanting & van der Plicht (1998) that routine measurement of $\delta^{15}N$ should be undertaken when human bone samples are submitted for ¹⁴C age measurement and should be regarded as an essential part of the 14C dating process for human bone samples from the Iron Gates.

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