

## RADIOCARBON DATING, STABLE ISOTOPE ANALYSIS, AND DIET-DERIVED OFFSETS IN $^{14}\text{C}$ AGES FROM THE KLIN-YAR SITE, RUSSIAN NORTH CAUCASUS

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**ABSTRACT.** The influence of geothermally derived carbon on the radiocarbon dating of human bone from archaeological sites is poorly understood and has rarely been rigorously examined. This study identifies a previously unknown reservoir effect at the archaeological site of Klin-Yar in the Russian North Caucasus. AMS-dated human bones yielded results that were older than expected when compared with dates of coins found in the same grave contexts. We investigated the reasons for this offset by AMS dating modern plant, fish, and water samples to examine the source of the old carbon. We identified a potential source in geothermally derived riverine and spring water, with an apparent age of several thousand years, and hypothesize that carbon from here is being transferred through the food chain to humans. If humans consume resources from the local rivers, such as fish, then they ought to be affected by this reservoir offset. An extensive analysis of carbon and nitrogen isotopes of human and animal bone showed evidence for a mixed diet that may be masking the amount of freshwater-derived protein being consumed. Due to the highly variable nature of the  $^{14}\text{C}$  offset (0 to ~350 yr), no suitable average correction factor is applicable to correct for the human dates at the site. A  $^{14}\text{C}$  chronology based on dates obtained from terrestrial ungulate bones, which we subsequently obtained, is instead a more reliable indicator of age.

### INTRODUCTION

One of the primary assumptions of radiocarbon dating is that the concentration of  $^{14}\text{C}$  within an organism is in equilibrium with that of the atmosphere. This is demonstrably not, of course, always the case.  $^{14}\text{C}$ -depleted carbon can enter the food chain causing  $^{14}\text{C}$  reservoir effects, which will cause dates to appear aberrantly old. The oceanic system provides the most commonly cited case. Here, ocean deep water cannot exchange carbon with the atmosphere and so, when it upwells to the surface and mixes, organisms feeding in the upper reaches of the ocean will appear, on average, around 400 yr too old (Berger et al. 1966; Mangerud 1972; Stuiver and Braziunas 1993; Reimer and Reimer 2001).

In freshwater systems, old carbon can be derived from numerous sources including dissolved limestone, peat, and geothermal vents in lakes (Lanting and van der Plicht 1998). Therefore, freshwater reservoir effects may be expected to differ between, and within, each river and lake. These effects have great potential to influence  $^{14}\text{C}$  dates of organisms living within and around these environments, including humans procuring resources from them. If reservoir effects are found in humans, however, they also enable us to detect the presence of freshwater resources, such as fish, within the prehistoric economy. These items are notoriously difficult to identify because fish skeletal evidence is exceptionally fragile and, where it does survive, is rarely recovered without careful excavation and sieving.

Paleodietary reconstruction using stable isotope analysis is based on the principle that different foods contain distinct isotopic signatures that are passed along the food chain to their consumers. Carbon and nitrogen stable isotopic analysis may provide an indication of the relative consumption of marine versus terrestrial (Tauber 1981) or terrestrial  $\text{C}_3$  versus  $\text{C}_4$  diets (Vogel et al. 1977). There are a number of complications to this, one of which is the inclusion of freshwater fish within the diet.

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Because of the numerous allochthonous sources of carbon coming into many freshwater systems, freshwater resources can have a wide range in stable isotopic values.

While much of this range overlaps with that expected in terrestrial organisms, high  $\delta^{15}\text{N}$  values or unusually enriched or depleted  $\delta^{13}\text{C}$  values, are sometimes held to indicate the presence of freshwater resources within a diet. This is not always the case, however. For  $\delta^{15}\text{N}$  values, freshwater systems generally have fewer trophic levels than marine food webs (Gannes et al. 1998) and can have correspondingly lower average  $\delta^{15}\text{N}$ . France's (1995) review of the isotopic values of 549 freshwater fish, for example, yielded a mean freshwater  $\delta^{15}\text{N}$  value of  $9 \pm 3\text{‰}$ . This may be compared to the estimates of Richards and Hedges (1999) of 8–22‰ for marine carnivores and 4–10‰ for terrestrial herbivores. While several stable isotope studies have identified the presence of freshwater fish within a population (e.g. O'Connell et al. 2003), the significant overlap between freshwater isotopes and terrestrial isotopes often precludes the routine application of stable isotopic markers.

$^{14}\text{C}$  offers an additional (radio)isotopic indicator to identify freshwater uptake in humans, when there is a reservoir offset. Cook et al. (2001) and Bonsall et al. (2002), for instance, considered the freshwater reservoir effect in  $^{14}\text{C}$  dating the Mesolithic-Neolithic transition sites of the Serbian Iron Gates region. They measured the  $\delta^{15}\text{N}$  values of organisms that exploited a 100% aquatic diet, and then compared them with the values of wholly terrestrial organisms. Using these values as end members, they plotted a linear relationship between  $\delta^{15}\text{N}$  values and the level of protein derived from aquatic protein. Higher  $\delta^{15}\text{N}$  values of human bones yielded significant offsets of 300–500 yr. They corrected the  $^{14}\text{C}$  dates by applying a reservoir estimate based on an interpolation of the  $\delta^{15}\text{N}$  data representing extreme freshwater and terrestrial values, and the expected offset in  $^{14}\text{C}$  years. This provided a more consistent age estimate than simply viewing the results at face value, although it has been criticized (Boric and Miracle 2004). Generally speaking, the freshwater reservoir effects remain a little studied, but potentially serious influence on the  $^{14}\text{C}$  ages of humans from Holocene sites (Lanting and van der Plicht 1998; Shishlina et al. 2007).

## THE SITE OF KLIN-YAR

Klin-Yar is a site in the Russian North Caucasus, near the city of Kislovodsk (Figure 1). It comprises a substantial sandstone ridge, on and around which prehistoric to early medieval settlements and cemeteries have been discovered (Figure 2). The largest cemetery (Klin-Yar III) spans 3 cultural phases, from the Late Bronze Age/Early Iron Age Koban culture to the early medieval Alans. Around 350 graves have been excavated thus far, but between 1000 and 3000 inhumations are estimated to be located in the site environs. The Bronze Age/Iron Age Koban inhumations at the site were composed of single burials in grave pits, often within a stone lining or stone covering. Males were buried on their right side, women on their left. Later Iron Age Sarmatian inhumations were usually interred in small underground chambers. In instances where double burials were excavated, the male was buried in a separate chamber to the female, but the 2 chambers were often linked by an entrance pit or corridor. Early medieval Alanic graves were more standardized in their layout and comprised a narrow dromos or passage leading towards an underground burial chamber. Often, within the passage, broken ceramics, charcoal, horse skin depositions, or complete horses with horse gear are found. Heads were orientated to the west and the body was interred extended on the back or slightly turned to one side. The discovery of an extremely rich Late Sarmatian-Alanic plot at Klin-Yar by two of us (AB and HH) has yielded several spectacular grave assemblages, many of them including whole horse depositions within what is interpreted as an elite area of the cemetery (Härke and Belinsky 2000).



Figure 1 Location of the site of the Klin-Yar, Russia. North is the top of the diagram.

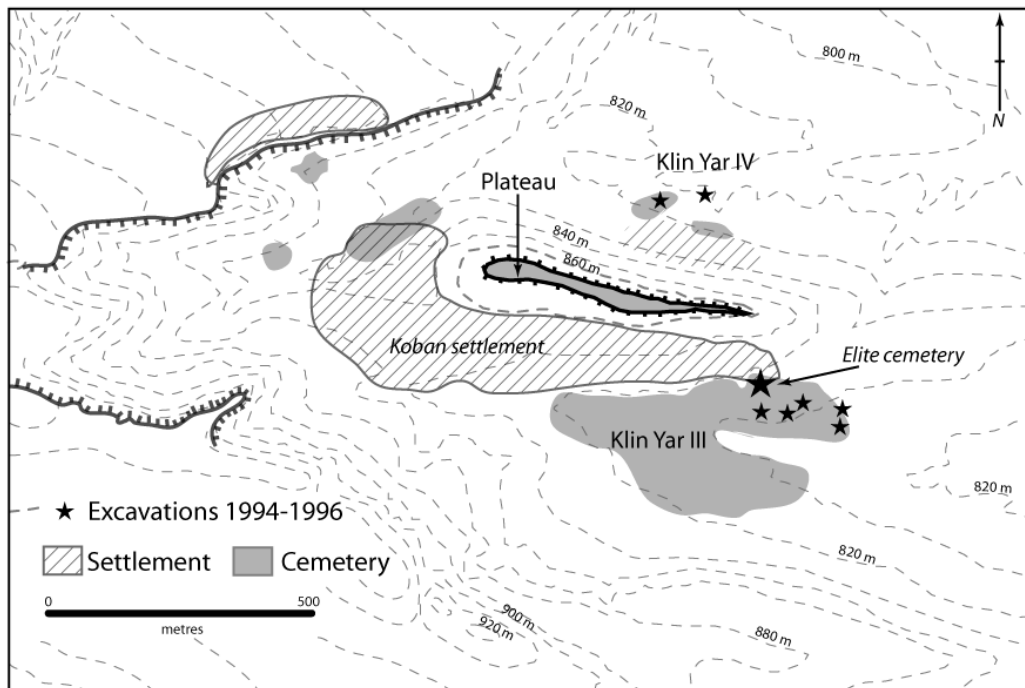


Figure 2 Excavation locations at Klin-Yar. The site is located around a large limestone plateau, surrounded by settlement and cemetery sites. The location of the elite cemetery is shown on the plan.

**RADIOCARBON DATING AT THE KLIN-YAR SITE**

An initial series of  $^{14}\text{C}$  dates was obtained from human bone excavated in 1994–96 to test the accuracy of dates derived from numismatic evidence and associated material culture. The  $^{14}\text{C}$  results yielded surprising offsets from the dates expected (Table 1 and see Bronk Ramsey et al. 2002:77–8). OxA-9388 dates Grave 363 at  $1507 \pm 38$  BP, or AD 534–611 (0.95% prob.) but a coin dating to the Byzantine Emperor Heraclius and his sons (AD 634–641) was found in the same context. The coin was part of a gold necklace placed into the grave and had been specifically pierced for the purpose. There is no doubt about its context and association to the burial remains. In grave 341, 2 coins of the Byzantine Emperor Maurice dating to AD 582–602 were found deposited probably in a bag with 2 earrings and a gold medallion, directly next to the skull, but the  $^{14}\text{C}$  age (OxA-9386) obtained was between 340 and 240 yr older. Artifacts in one of the other graves (360) included jewelry, a mirror, an Iranian glass vessel, a sword with characteristic fittings, an iron stirrup, and 2 pottery vessels supporting a date in the first half/middle 7th century AD. Again, the associated  $^{14}\text{C}$  dates are much older than expected. In total, the offsets between the ages inferred using relative and numismatic evidence and the measured AMS determinations ranged from almost nothing to several hundred years (Table 1). We measured the offsets as probability distributions by determining the difference between the  $^{14}\text{C}$  and historic ages using OxCal v 4.1 (Bronk Ramsey 2009) and accounting for the uncertainties on the latter ages (the probability offsets for each dated human bone are plotted in Figure 3a).

Table 1 Initial  $^{14}\text{C}$  ages obtained from the Klin-Yar site. Historic dates and date ranges are shown in column 7. Stable isotope values are reported with respect to VPDB (carbon) and AIR (nitrogen). Measurement errors are  $\pm 0.2\%$  for carbon and  $\pm 0.3\%$  for nitrogen. Results are calibrated against the IntCal04 terrestrial data set (Reimer et al. 2004). Offsets are calculated using OxCal 4.1 (Bronk Ramsey 2009). (Differences between historic cal date and  $^{14}\text{C}$  age BP are at 68.2% probability ranges unless specified.)

OxA nr	Sample reference/ Burial number	$\delta^{13}\text{C}$ (‰)	$\delta^{15}\text{N}$ (‰)	$^{14}\text{C}$ age BP	Calibrated age range (1 $\sigma$ ) (95.4% probability in brackets)	Estimated historic date AD	Offset from historic age
9388	KY III 363B/2 – Human bone	–17.3	10.3	$1507 \pm 38$	AD 470–477 (0.04) AD 534–611 (0.95)	634–641 [Coin]	–103–25 (63.8%)
9380	KY III 363A – Bone of a child scattered at entrance to catacomb 363B	–15.7	10.2	$1960 \pm 45$	36–31 BC (0.04) 0–12 BC (0.064) AD 1–82 (0.892)	Earlier than 634–641 [Coin]	n.a.
9386	KY III Burial 341/1 – Human tooth	–16.8	9.9	$1741 \pm 39$	AD 243–344 (1.00)	582–602 [Coin]	–343–240
9387	KY III Burial 341/3 – Human tooth	–17.6	10.6	$1568 \pm 37$	AD 434–494 (0.675) AD 505–538 (0.325)	582–602 [Coin]	–150–50
9389	KY III Burial 378 – Human tooth	–13.1	9.3	$1879 \pm 38$	AD 74–140 (0.727) AD 150–170 (0.152) AD 194–210 (0.122)	Late 4th–early 5th C	–309–209
9390	KY III Burial 360/1 – Human tooth	–16.7	11.3	$1685 \pm 37$	AD 264–275 (0.098) AD 333–410 (0.90)	Early-mid 7th C	–297–180
9391	KY III Burial 360/2 – Human tooth	–16.5	10.3	$1556 \pm 39$	AD 434–494 (0.625) AD 505–523 (0.160) AD 526–546 (0.215)	Early-mid 7th C	–162–50

There are several reasons we might propose as to why such offsets may occur. These include: 1) contamination of the bone with exogenous carbon; 2) a reservoir effect derived from the uptake of protein from marine sources; or 3) old carbon from a mineral source entering a freshwater system, being fixed by aquatic plants and entering the food chain, ultimately ending up in humans.

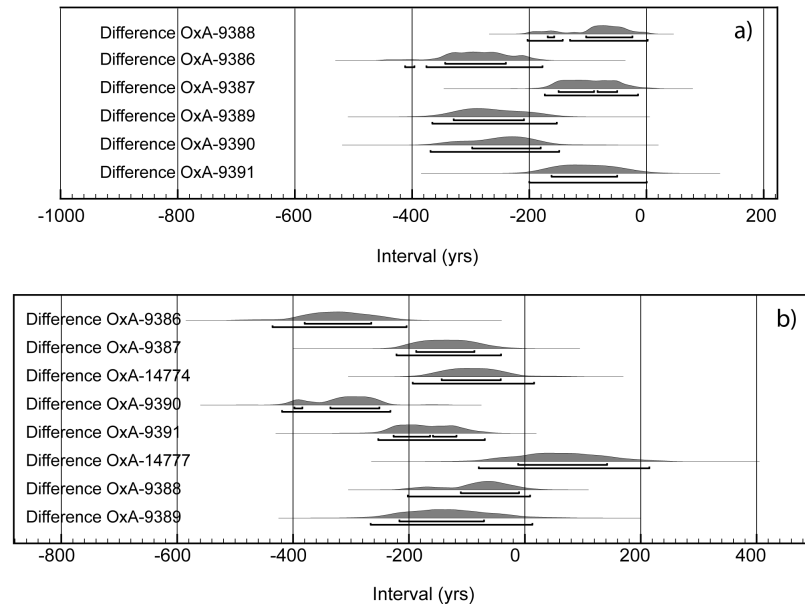


Figure 3 a) Human bone <sup>14</sup>C offsets from historic age, as deduced from material culture and numismatic evidence (see Table 1 for data). b) AMS date offsets as deduced from comparison with animal bone from the same contexts (see Table 2 for data). The offsets are plotted as probability distributions using OxCal 4.1 (Bronk Ramsey 2009).

The first possibility, that of contamination, is thought unlikely to be the explanation. The bones were reasonably well preserved and the analytical data obtained (e.g. C:N ratios, stable isotopes, %carbon, %collagen, etc.) disclosed no significant problems, although it should be acknowledged that none of these methods individually are precise indicators of contamination. The marine reservoir effect is also unlikely to be the explanation. Klin-Yar is about 150 miles from the Black Sea and 300 miles from the Caspian Sea. Maintaining what would need to be a fairly constant supply of marine fish across such distances would have taken a considerable amount of both energy and time, and must therefore be deemed unlikely. In addition, the nitrogen isotopic data, averaging ~10.0‰, are not indicative of a substantial marine fish influence. The values would be expected to be somewhat higher than this for a significant marine component, although quantification is difficult (Hedges 2004).

Old carbon entering the food chain is a more likely possibility. It is widely known that there is extensive geothermal activity in the environs of Klin-Yar. Carbonaceous spring waters locally are bicarbonate anion predominant. Geothermal sources have been observed to introduce <sup>14</sup>C-depleted CO<sub>2</sub> into local environments (including bodies of water) in other regions, producing apparent ages for living organisms within or adjacent to the source (Bruns et al. 1980; Rubin et al. 1987; Pasquier-Cardin et al. 1999; Beavan-Athfield et al. 2001). For old geothermally derived carbon to affect the <sup>14</sup>C dates of human bones from the Klin-Yar site, the old carbon has to be fixed at the base of the food chain in aquatic plants and passed on through it, ending up in humans. The most likely source of transfer to humans might be through the consumption of freshwater resources from the local rivers, principally the Podkumok, which runs near the site. However, there is no archaeological evidence for the exploitation of fish at Klin-Yar. This mirrors a general absence of fish in sites dating to the later Bronze Age and early Iron Age in this region. Klin-Yar was excavated in 1994–96 and for technical and equipment reasons, flotation and small screening was not possible. Two fossil mollusks were excavated (in

Alanic graves 345 and 374); therefore, if mollusk shells were present in graves they would have been discovered. It may be that with flotation, small fish remains might have been recovered.

Klin-Yar may therefore present an interesting scenario within which to examine the relationship between archaeological evidence, stable isotopic data, and freshwater  $^{14}\text{C}$  reservoir effects as indicators for the consumption of resources from freshwater sources. The first aim of this study was to examine whether the offset seen between the age of human bones and artifactual evidence could be clarified by further dating work. As shown above, the  $^{14}\text{C}$  age of human bones can be compared to well-dated artifacts, and also be compared with the ages derived from terrestrial ungulate bones excavated in close association (in grave fill and near skeletal remains). In the absence of ancient fish remains, and to test the hypothesis that the age of carbon within the freshwater is affected by geothermal springs, modern river water and fish were collected and dated. Finally, stable isotope analysis was undertaken on further samples of human and faunal bone to examine whether this technique might be able to provide further evidence concerning fish consumption during the period and to shed further light on paleodiet and economy during this period of Caucasus prehistory and early history.

## MATERIALS AND METHODS

Bones of terrestrial ungulates (horse and cow), as well as humans, from some of the graves were selected from collections at Nasledie in Stavropol and the Institute of Archaeology, Russian Academy of Sciences (Rossiskaya Akademiya Nauk, RAN), Moscow. Some of the animal bone came from graves that had been dated previously using human bone (Table 1), to examine whether ungulates were also subject to a reservoir offset and, if possible, to quantify the extent of such an offset in the dated human bone. In almost every case, the animal bones selected were part of deliberate depositions of material within the grave context. Stable isotopes of carbon and nitrogen were analyzed separately and selected from a wider range of human and animal bones.

Bone for this study was either sampled using tungsten carbide drills or as a  $\sim 1 \times 2$ -cm section of bone, cut using a diamond saw. These samples were cleaned at ORAU by scraping with a scalpel to remove soil coloration, sediment detritus, and weathered surfaces, before being shot-blasted using aluminium oxide. Whole bone samples were powdered in a mortar and pestle. Samples were pre-treated at the ORAU using previously published routine methods. Bone powder samples were treated with a sequence of hydrochloric acid (0.5M) to decalcify the bone, then sodium hydroxide (0.1M), and hydrochloric acid (0.5M) with thorough rinsing with ultrapure (Milli-Q<sup>TM</sup>) water between each reagent (Bronk Ramsey et al. 2004). They were then gelatinized in weakly acidic water (pH 3) at 75 °C for 20 hr, with the supernatant recovered using an EziFilter<sup>TM</sup>. Each sample for  $^{14}\text{C}$  dating was ultrafiltered using the methods outlined in Bronk Ramsey et al. (2004) and Higham et al. (2006). Samples for stable isotope analysis were not ultrafiltered, nor were they base washed, following the protocol of Richards and Hedges (1999).

All samples were weighed into precleaned tin capsules and then combusted. The gas was analyzed using a Europa Scientific ANCA-MS system consisting of a 20–20 IR mass spectrometer interfaced to a Roboprep CHN elemental analyzer operating in continuous-flow mode using an He carrier gas. This enables the measurement of  $\delta^{15}\text{N}$  and  $\delta^{13}\text{C}$ , nitrogen and carbon content, and C:N atomic ratios.  $\delta^{13}\text{C}$  values for  $^{14}\text{C}$  measurements cited in this paper are reported with reference to VPDB (Coplen 1994) and  $\delta^{15}\text{N}$  results are reported with reference to AIR. For samples to be dated, graphite was prepared by reduction of  $\text{CO}_2$  over an iron catalyst in an excess  $\text{H}_2$  atmosphere at 560 °C prior to AMS  $^{14}\text{C}$  measurement (Bronk Ramsey and Hedges 1999; Bronk Ramsey et al. 2000). Wherever gelatin yield permitted, stable isotope analyses were measured in triplicate.

A small number of modern plants were collected near the Klin-Yar site and from the margins of a spring ~100 m from it by TH. In addition, plants and water were collected from the Podkumok River, near the town of Tereze. The aim was to measure the <sup>14</sup>C concentration and determine whether or not these materials differed significantly when compared with modern atmospheric values. A modern fish (*Salmo trutta*) was also obtained from the same river.

## RADIOCARBON RESULTS

The <sup>14</sup>C ages obtained from paired horse and cow bones in the same burial context are shown in Table 2. The results are compared with the human bone determinations from the same grave contexts. We assume that the ages for the terrestrial ungulate bones are equivalent to the ages that would be expected for the associated human bones in the absence of a reservoir effect. This assumption is not always justifiable, of course, since grave contexts such as these were sometimes looted in the past, resulting in the redeposition of some material. Similarly, archaeological contexts are often subjected to the gamut of site formation processes that influence the original association between objects. In the vast majority of cases, however, we were confident that this was of minimal importance. As previously mentioned, the 3 coins found were in deliberate grave depositions and the dated animal bones were carefully excavated from within grave contexts. All of the horse bones, for instance, were derived from entire horses deposited in access corridors (dromos), or from “horse skins” (heads-and-hooves depositions) on or in corridors, or next to a corridor. All of these remains were found in articulation. The context of these bones is therefore unimpeachable. The only exceptions from the rule of careful deposition within a ritual context are Alanic graves 357 and IV-9 (previously burial 4) where the burial chamber contents had been cleared out into the access corridor (dromos) in antiquity, but even here it is reasonable to assume that any animal bones found originated from the chamber or the dromos itself. The results for the cow bone in grave 361A, however,

Table 2 Comparison between <sup>14</sup>C ages of animal (horse/cow) and human bone from the same archaeological context. These are calibrated using the IntCal04 curve (Reimer et al. 2004). Offsets between the human and associated ungulate bones are shown in column 9. Offsets are calculated using OxCal as described in the text and Table 1. No offset is calculated for Grave 361A due to the obviously intrusive nature of the cow bone in the grave context.

OxA Nr	Sample reference	$\delta^{13}\text{C}$ (‰)	$\delta^{15}\text{N}$ (‰)	C:N ratio	<sup>14</sup> C age BP	Calibrated age range AD (68.2% prob.)		<sup>14</sup> C offset (yr)
						From	To	
9386	KY III 341/1 – Human	–16.8	9.9		1741 ± 39	242	345	–380–265
9387	KY III 341/3 – Human	–17.6	10.6		1568 ± 37	434	538	–187–87
14839	KY III 341 – Horse	–20.2	5.7	3.2	1428 ± 28	608	648	
14774	KY III 357 – Human	–17.3	11.7	3.3	1582 ± 28	431	533	–143–41
14773	KY III 357 – Horse	–19.6	6.0	3.3	1497 ± 28	547	600	
9390	KY III 360/1 – Human	–16.7	11.3		1685 ± 37	264	410	–397–251
9391	KY III 360/2 – Human	–16.5	10.3		1556 ± 39	434	547	–226–118
14885	KY III 360 – Cow	–19.7	5.1	3.5	1356 ± 28	650	685	
14886	KY III 360 – Horse	–20.3	4.9	3.6	1345 ± 29	649	674	
14775	KY III 361A – Human	–16.9	11.2	3.1	1798 ± 30	139	254	n.a.
14776	KY III 361A – Cow	–18.8	7.5	3.2	2981 ± 31	–1268	–1131	
14777	KY III 361B – Human	–15.2	11.4	3.2	1728 ± 38	254	379	–11–142
14840	KY III 361B – Horse	–20.2	3.8	3.2	1780 ± 30	214	326	
9388	KY III 363B/2 – Human	–17.3	10.3		1507 ± 38	471	611	–110–10
14047	KY III 363B – Horse	–20.1	4.3		1411 ± 28	616	654	
9389	KY III 378 – Human	–13.1	9.3		1879 ± 38	74	210	–216–70
14048	KY III 378 – Horse tooth	–20.4	4.1		1771 ± 28	230	326	

are a reminder of the potential dangers in assuming contemporaneity of non-horse material in certain burial locations. This bone would seem to have originated from the earlier Koban settlement on this site, or be from a disturbed Koban burial, based on the  $^{14}\text{C}$  date.

The offset between the human and animal bone determinations ranges up to several centuries (Table 2 and Figure 3b), which parallels the range shown earlier in Table 1. Comparison of the  $^{14}\text{C}$  offset data with the stable isotope results yields no strong correlation (Figures 4 and 5); wide differences in offset are not linked with stable isotope values.

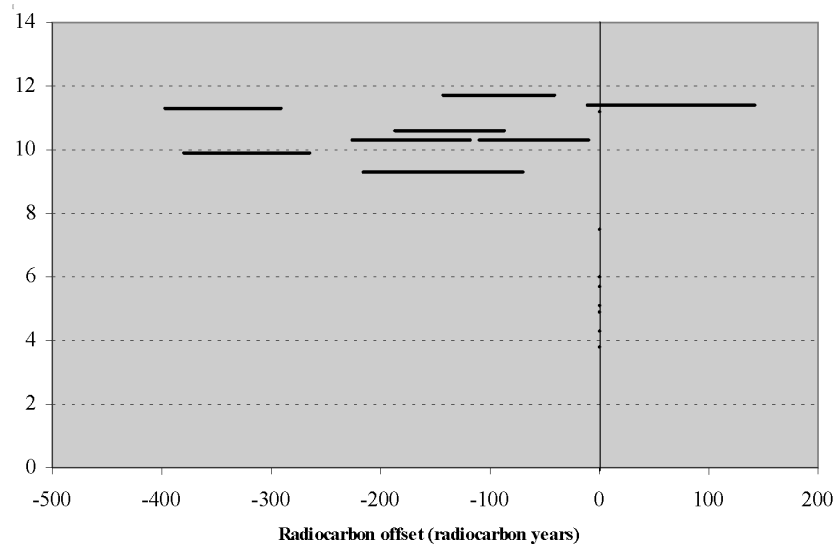


Figure 4 Relationship between  $\delta^{15}\text{N}$  and the offset in  $^{14}\text{C}$  yr between human and animal bones from the same grave context (data from Table 2).

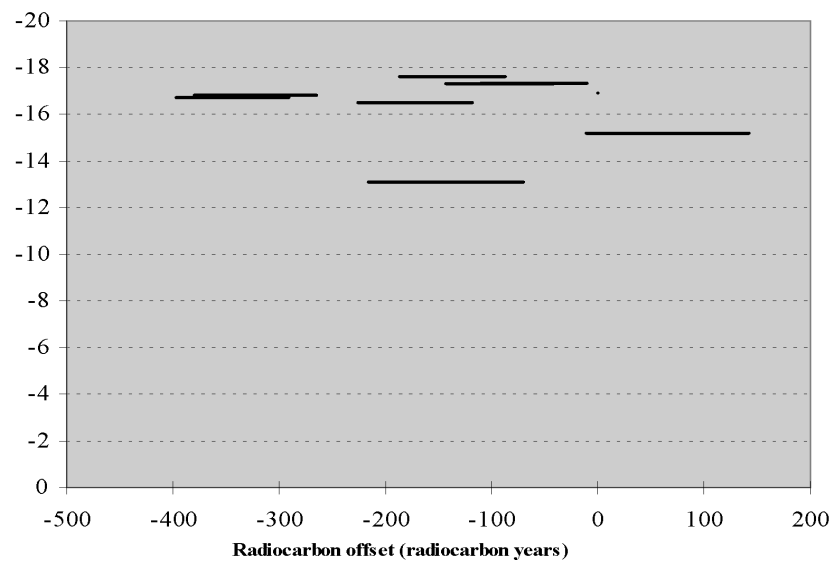


Figure 5 Plot of  $\delta^{13}\text{C}$  and offset in  $^{14}\text{C}$  yr between human and animal bones from the same grave context (data from Table 2).



Results for the modern samples of fish, water, and plants are shown in Table 3. The <sup>14</sup>C values for grass and aquatic plants within the immediate vicinity of the Klin-Yar site are close to those expected, based on comparisons with current atmospheric pMC values measured at ORAU, for modern plant matter. The most depleted result was for water taken from the Podkumok River, at  $66.0 \pm 0.3$  pMC. Unfortunately, the amount of carbonate extracted from the water varied between repeat analyses; therefore, this measurement may not be wholly accurate. Despite this, the substantially depleted pMC measurement does suggest strongly that there is a significant offset between water and atmosphere. Taken together, the values suggest that there is old carbon in these waters diluting the <sup>14</sup>C ages of organisms living within the reservoir. Although calculation of a precise offset is problematic due to the bomb carbon effect, we estimate a reservoir effect of ~800 yr for the Tereze aquatic plant and ~600 yr for the river fish.

Table 3 <sup>14</sup>C pMC values for modern collected (June 2005) plant, water, and fish samples. Tereze samples come from the Podkumok River. The samples from Klin-Yar came from on top of the limestone plateau at the site and ~100 m away from a spring (see text). An OxA-X value denotes a date of less certainty. The HCO<sub>3</sub> yield from the Tereze water was low and variable between extractions, meaning that this age should be viewed with some caution in terms of its accuracy. The water value mirrors DIC activities from similar spring waters undertaken by Marčenko et al. (1989) at the Plitvice Lakes in Croatia, which ranged between 60 to 84 pMC, while atmospheric CO<sub>2</sub> activity changed from 130 pMC (1984) to 120 pMC (1988).

OxA/OxA-X nr	Material	δ <sup>13</sup> C	δ <sup>15</sup> N	pMC value
14860	Klin-Yar <i>Poa</i> sp. ( <i>Poaceae</i> family) grass	-26.3	-1.9	105.5 ± 0.3
14859	Klin-Yar aquatic plant ( <i>Juncus</i> sp.) Rush	-29.0	6.1	106.0 ± 0.3
14882	Tereze River aquatic plant matter (not identified)	-19.8	8.4	94.6 ± 0.3
14861	Tereze Trout ( <i>Salmo trutta</i> ) flesh	-26.7	11.7	97.5 ± 0.3
-X-2139-18	Tereze Water HCO <sub>3</sub>	-19.7	nd	66.0 ± 0.3

## STABLE ISOTOPE RESULTS

The results of carbon and nitrogen stable isotope analyses are summarized in Table 4 and shown in Figure 6. The human stable isotope data is plotted with respect to cultural attribution; Koban, Sarmatian, or Alanic, although some amongst the Sarmatian group may be attributed to a transitional Sarmatian/Alanic phase. It can be seen that the range in δ<sup>13</sup>C among the humans is significant and covers ~6‰. Koban humans appear to range between about -13 and -16‰ and, therefore, appear to be more enriched in δ<sup>13</sup>C when compared with individuals from both Sarmatian and Alanic contexts. Sarmatian values also disclose a wide range. The values obtained are not normally distributed and disclose some skewness (see Figure 7). To explore this further, we applied a series of non-parametric statistical tests of the data. First, we applied a Kruskal Wallis test, with a null hypothesis that the multiple independent variables are from same population. This is a one-way analysis of variance by rank and tests the relatedness of population medians amongst groups of data. The analysis showed that the samples are indeed from different populations. To determine which of the 3 populations were statistically different, we applied a Mann Whitney U test, which allows an assessment of whether 2 independent samples of observations come from the same distribution (where  $H_0$ ; 2 independent samples come from the same group). This showed that the Koban population is different to the Sarmatian and Alanic groups, but that these latter 2 are indistinguishable from one another (if  $p < 0.05$  we can reject  $H_0$ ) (the Sarmatian and Alanic populations are statistically indistinguishable [ $Z = -1.24$ ,  $p = 0.22$ ] while the Sarmatian and Koban [ $Z = -4.38$ ,  $p = 0.00$ ] and Alanic and Koban [ $Z = -4.23$ ,  $p = 0.00$ ] populations are different).

Table 4 Stable isotope values for humans from Klin-Yar. Sex and age identifications are based on Buzhilova et al. (in press).

	Age/Sex	$\delta^{13}\text{C}$	Std dev	$\delta^{15}\text{N}$	Std dev
<b>Alanic</b>					
237	nd.	-18.3	0.23	9.9	0.1
382/3	Child, 2–3 yr	-14.4	0.05	11	0.03
352/1 Elite	?adult	-18.3	0	11.2	0.04
359 Elite	Female, 25–29 yr	-18.6	0.01	9.9	0.09
360/1 Elite	Male, 35–45 yr	-18.7	0.06	11.3	0.08
363B/1 Elite	Female, 30–34 yr	-17.8	0.05	11	0.12
364 Elite		-18.4	0.03	10.9	0.05
368 Elite	Male, 35–45 yr	-17.9	0.08	10.4	0.14
371/1 Elite	Female, 20–25 yr	-18.6	0.01	10.2	0.04
373 Elite	Female, 25–29 yr	-18.1	0.06	9.9	0.02
374 Elite	Juvenile, 15–19 yr	-17.6	0.02	10.9	0.11
380 Elite	Infant, 1.5 yr $\pm$ 6 months	-17.2	0.04	15.9	0.07
381/1 Elite	Male, 20–25 yr	-17.6	0.05	10	0.05
<b>Sarmatian</b>					
235	nd.	-18.2	0.02	10.2	0.05
383 (transitional Sar/Alans)	Child, 7–14 yr	-17.3	0.11	11.9	0.06
385	Juvenile, 10–13 yr	-18.3	0.06	10.2	0.1
387 (transitional Sar/Alans)	Male, 40–49 yr	-18.5	0.03	10	0.05
342B	Female, 30–40 yr	-17.4	1.12	9.8	0.48
351A Elite	Male, 20–24 yr	-15.6	0.03	9.1	0.03
351B Elite	Female, 30–40 yr	-16.6	0.1	10.2	0.02
361A Elite	Female, 20–25 yr	-17.5	0.32	10.5	0.27
361B Elite	?Female, 20–29 yr	-15.7	0.04	10.0	0.11
370 Elite	Female, 25–30 yr	-18.1	0.08	9.6	0.02
372 Elite	Male, 35–45 yr	-18.3	0.06	9.9	0.1
378 Elite	Child, 10–12 yr	-14.4	0.12	9.6	0.07
379 Elite	Female, 25–30 yr	-18.4	0.14	10.1	0.14
386/2	Female, 25–29 yr	-18.2	0.02	10.2	0.05
<b>Koban</b>					
11	nd.	-14.6	0.04	11.4	0.12
172		-15.5	0.04	11.2	0.02
181	Female	-15	0.06	11.3	0.22
187		-15.8	0.01	10.9	0.18
189	Female	-14.6	0.1	10.6	0.12
190		-16	0.05	10.2	0.09
193		-15.5	0.03	10.9	0.22
206	Male	-14.4	0.1	11.1	0.04
207		-15	0.03	9.63	0.17
208		-15	0.1	10.6	0.05
211		-15.8	0.18	10.9	0.07
214		-14.8	0.06	10.5	0.12
220	Male	-14.1	0.07	10.5	0.05

Table 4 Stable isotope values for humans from Klin-Yar. Sex and age identifications are based on Buzhilova et al. (in press). (Continued)

	Age/Sex	$\delta^{13}\text{C}$	Std dev	$\delta^{15}\text{N}$	Std dev
236		-15.4	0.12	11.1	0.14
241	Male	-15.9	0.03	11.5	0.08
277		-14.3	0.07	10.9	0.08
285		-14	0.05	10.2	0.04
315		-14.6	0.04	11	0.03
320		-13.4	0.04	10.7	0.04
323		-14.9	0.1	10.6	0.08
324	Male	-14.6	0.15	10.4	0.19
349	Male, 30–40 yr	-14.3	0.04	10.9	0.08
355	Male, 40–50 yr	-16	0.03	12	0.02
377	Child, 9–10 yr	-14.6	0.16	10.8	0.15

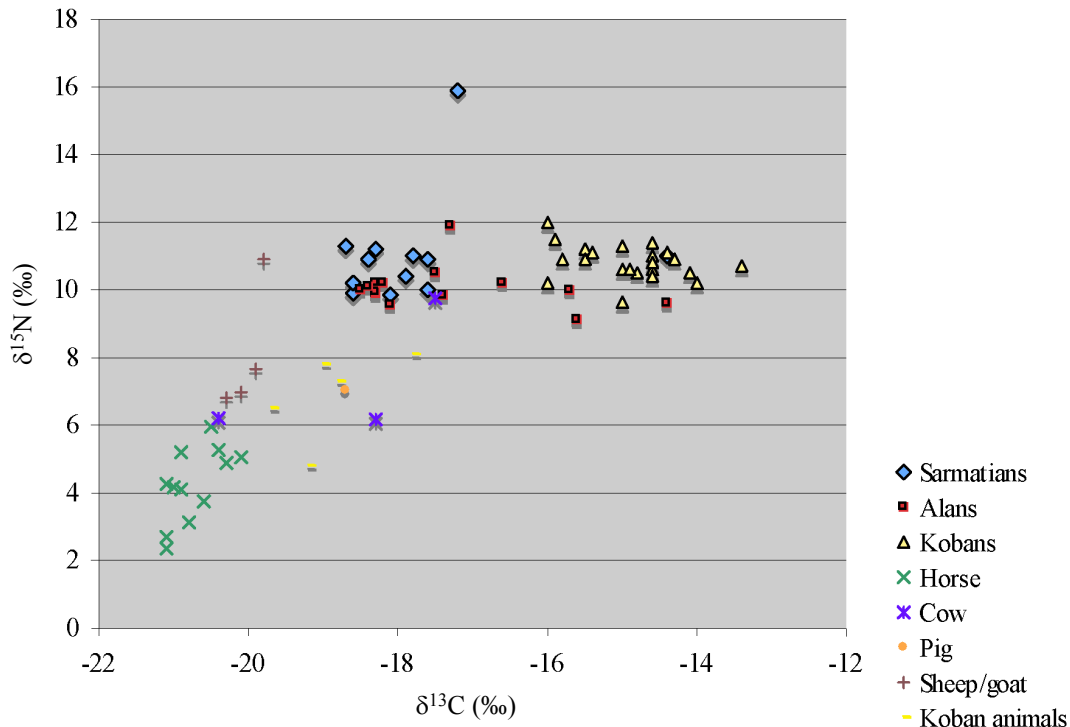


Figure 6 Stable isotope values for humans and animals. Human values are divided by cultural attribution. Animal values are all associated with Alanic and Sarmatian groups, whereas results from animal bones associated with the Koban settlement are all given in yellow. The highest human value is for an infant probably being breastfed. Breastfeeding infants are well known to be enriched in  $\delta^{15}\text{N}$  by 2–3‰ over their mothers' values, since they are essentially a trophic level higher (Fuller et al. 2003). Note that this plot includes only values measured in specific diet analyses, not the AMS-dated samples given in Table 1. Data are given in Tables 4 and 5.

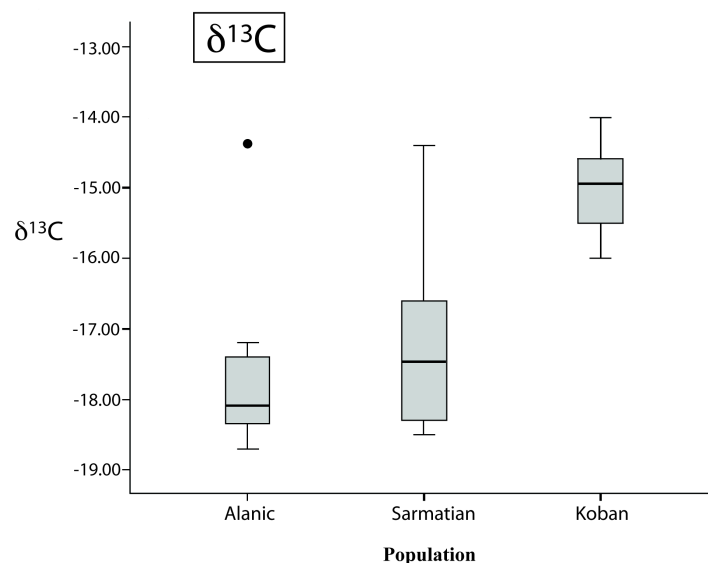


Figure 7 Distributions of the  $\delta^{13}\text{C}$  values obtained for Alanic, Sarmatian, and Koban populations. The results show the skewness of the Alanic and Sarmatian groups.

We have also considered the distribution of  $\delta^{15}\text{N}$  values obtained for the Koban, Sarmatian, and Alanic populations. Once again, a degree of skewness and non-normality was noted in the data set (Figure 8). Non-parametric tests showed that Koban and Alanic groups are indistinguishable (Mann Whitney U test,  $Z = -1.39$ ,  $p = 0.17$ ) and that the Alanic and Sarmatian groups, too, are the same statistically ( $Z = -1.98$ ,  $p = 0.05$ ). The Sarmatian and Koban groups, however, do appear statistically different ( $Z = -4.41$ ,  $p = 0.00$ ), but the differences here are very small ( $<1\%$ ) and more data points are required to add strength to the conclusions.

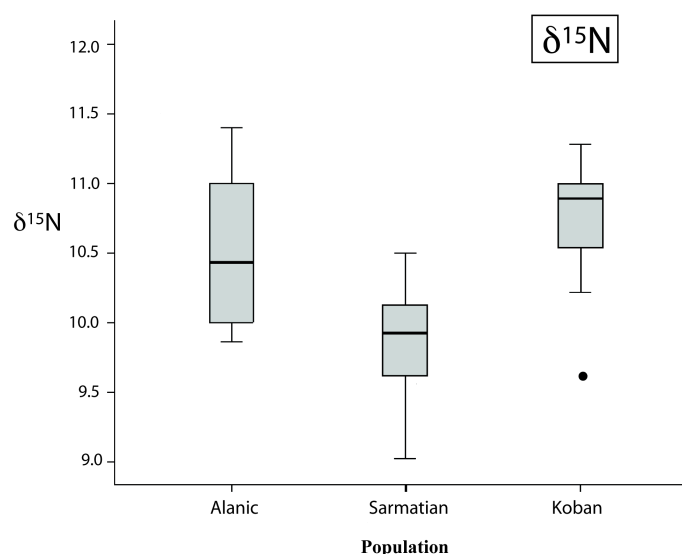


Figure 8 Distributions of the  $\delta^{15}\text{N}$  values obtained for Alanic, Sarmatian, and Koban populations showing once again a degree of skewness and non-normality.

The results show, then, that there is a significant difference in the  $\delta^{13}\text{C}$  values of the Koban group on the one hand, and the Sarmatian-Alanic group, on the other, and that there is a wide range in carbon isotope results amongst all groups, but particularly the Sarmatian and Alanic individuals. Why is this? One possibility is that the Koban, and some of the Sarmatian/Alanic individuals, may be consuming protein originating via a  $\text{C}_4$  pathway. This could either be from 1) grain-based foods or 2) stock fed either from  $\text{C}_4$  grain or wild  $\text{C}_4$  grasses.

For the first possibility, the most likely candidate for a grain-based  $\text{C}_4$  crop would probably be millet (*Panicum miliaceum*). Millet is attested in the region substantially prior to the occupation date of the site, therefore it is not unreasonable to suggest its presence at Klin-Yar at a much later period. Lisitsina (1984; in Nesbitt and Summers 1988), for instance, reports the presence of “*Panicum* sp.” from Chokh in the Caucasus, dating from the beginning of the 6th millennium BC (see also Iacumin et al. 2004), although this is much earlier time period than the sites under discussion here. Millet is a species with a short growing season, high yields and has adaptability to a range of climates and soils (Tafari et al. 2009), so it may have been an attractive crop to grow in the Caucasus. More ethnobotanical work is clearly required.

For the second possibility, one has to consider the question of the distribution of  $\text{C}_4$  grasses and plants within the Caucasus that could have been used as grazing food for stock. Climate and environment are the key influences, because  $\text{C}_4$  plants are well adapted to minimize water losses due to photorespiration and, therefore, survive better in intense sunlight and higher temperatures. Klin-Yar is located in the forest-steppe region that follows the Kuban and Terek River systems. The boundary between the forest-steppe and the forest zones represents the boundary between more arid and more humid regions. To the northeast, the drier steppe region includes halophilous plant species, which include a predominance of  $\text{C}_4$  plants, but the major regional presence of these types is on the coastline of the northern Caspian Sea. Rainfall across this region varies too—in the Klin-Yar area, around 400–600 mm per annum (Volodicheva 2002)—but the region is characterized by dramatic changes in relief, temperature, and rainfall. Within 100 km of Klin-Yar, rainfall levels can be twice as high. Hollund et al. (2009) found that variations in both carbon and nitrogen isotopes probably originate from this environmental complexity in the North Caucasus (see also van Klinken et al. 2000).

Broadly speaking, through the Holocene,  $\text{C}_3$  plants dominated the North Caucasus region (Khokhlova et al. 2007), but it is possible that more aridity might have led to increased amounts of  $\text{C}_4$  grasses, which may be reflected in animal and, therefore, human stable isotope values. Khokhlova et al. (2007) analyzed carbon isotopes from paleosols in Alanic burial kurgans to examine paleoclimates in the northern Caucasus during the 1st to 5th centuries AD; their results suggest some climatic variability through the period. They found that from the end of the 2nd to the start of the 3rd centuries AD, values indicated clear xeromorphic properties indicative of a relatively dry climate with estimated rainfall around 50–100 mm less than today. They showed the return of these drier conditions in the mid-5th century AD. It is also possible that stock management practices may have involved wider geographic distances, taking in regions further from Klin-Yar where  $\text{C}_4$  grasses might have been more common. One means of shedding light on this would be to examine the stable isotope data from animal bones associated with the Koban-period settlement of Klin-Yar. Unfortunately, we obtained few well-contexted animal bones with which to test this suggestion, but interestingly those that were analyzed appear to be slightly more enriched in  $\delta^{13}\text{C}$  than corresponding Sarmatian-Alanic period animals, although only by 1–2‰ over non-Koban period animals. The data set is too small to draw confident conclusions (Figure 6) and more work is required. The present evidence suggests that it is more likely that the  $\text{C}_4$  signal is coming from millet, and that while millet may have been consumed by humans, it was not consumed to the same extent by stock. Further work

is required to examine more carefully the range of potential plants across this region, both from archaeological and geological sites. New ethnobotanical data would also be useful. Unfortunately, this lies outside the scope of the current work.

The wide range in the Sarmatian values particularly is intriguing. It may indicate that the population interred at Klin-Yar is made up of people who have migrated from different regions where different dietary preferences predominate and indeed this is the prevailing thought among specialists of the period.<sup>6</sup> An alternative explanation is that it represents the broad variety in local dietary regimes at the site, with a mixed  $C_4/C_3$  signal contributing to the wide range in stable isotopes. This could correlate with status. In the Sarmatian group, there is wide variation amongst all individuals, regardless of their inferred status in prehistory. For the Alanic burials, there is less variation amongst the group, and almost no non-elite burial data to compare against (Table 5). This aspect of the stable isotopic data is an interesting avenue of future research. We suggest that the variation between the different groups could be a reflection of very different economic adaptations. Koban groups are known to have been sedentary and agriculturally based, while Sarmatians were much more mobile and pastoralist in their economic approach. Differences between the groups, therefore, could simply be caused by access to very different food resources across widely different landscapes. Finally, of course, the ranges could represent temporal changes in diet, something that can only be explored with further careful dating.

Interpretation of the nitrogen isotopic data is difficult for a variety of reasons. It is widely known that nitrogen isotopes are enriched with successively increasing trophic levels, and that the values broadly reflect the average isotopic values of the dietary protein consumed by the individual animal or human. A human eating only plant protein, therefore, would yield a  $\delta^{15}N$  value the same as a local herbivore. There is a ~3‰ enrichment in  $\delta^{15}N$  values with successive trophic levels in the food chain (Schoeninger and DeNiro 1984), but recent work suggests there is potentially more complexity within this, and increasing levels of enrichment (up to about 5‰) have been suggested (Hedges and Reynard 2007). There is a lack of understanding of the influence of high- and low-protein diets on isotopic trophic enrichment. Another significant influence is climate and rainfall (van Klinken et al. 2000). The Caucasus is a region of dramatic variability in climate and environment, factors that strongly influence  $\delta^{15}N$  values plant and animal values. Unfortunately, little work has been undertaken in mapping this variation amongst plants in the region.

The presence of freshwater fish in human diets has often been detected on the basis of unusually high  $\delta^{15}N$  values in bulk collagen, as mentioned earlier, because the fish favored by humans are commonly carnivorous species. In the light of the uncertainty over enrichment magnitude and of the  $\delta^{15}N$  range of local freshwater fish, however, this is a potentially problematic area of interpretation. O'Connell et al. (2003) and Privat (2004) attributed enriched  $\delta^{15}N$  values in Bronze Age and Iron Age human bones from the Eurasian steppes to fish consumption. Lillie and Richards (2000) identified depleted  $\delta^{13}C$  values and enriched  $\delta^{15}N$  values with  $^{14}C$  reservoir effects, and implicated fish consumption amongst Mesolithic/Neolithic communities on the Dnepr River. Privat's (2004) data showed that average human  $\delta^{15}N$  ratios averaged ~12.0‰, while associated faunal herbivore values were 7‰. This difference of ~5.0‰ was taken as evidence of fish consumption because it is greater than the magnitude of the trophic level effect one usually expects. Again, the archaeological record was silent in terms of evidence for fish consumption; therefore, this conclusion has been controversially received (see also Shishlina et al. 2007).

<sup>6</sup>Sarmatians are thought to have migrated from the Volga region, while the Alans appeared from the central north Caucasus, ~200 km east of Klin-Yar.

Table 5 Stable isotope values for animals from Klin-Yar.

Koban to Alanic cemetery	$\delta^{13}\text{C}$	Std dev	$\delta^{15}\text{N}$	Std dev	C:N
347 Horse (Alanic)	-20.4	0.1	5.27	0.07	3.2
357 Horse (Alanic)	-20.1	0.05	5.06	0.22	3.2
359 unidentified (Alanic)	-20.4	0.08	7.41	0.12	3.2
360 Horse (Alanic)	-20.3	0.03	4.89	0.06	3.2
360 Pig (Alanic)	-18.7	0.02	7.02	0.1	3.2
363 Cow (Alanic)	-17.5	0.05	9.76	0.08	3.2
363 Horse (Alanic)	-20.8	0.03	3.12	0.08	3.2
366 Cow (Koban)	-18.3	0.03	6.18	0.06	3.2
367 Horse (Alanic?)	-21.1	0.04	2.36	0.02	3.2
370 Horse (Sarmatian)	-21	0.05	4.16	0.07	3.2
371 Horse (Alanic)	-21.1	0.11	2.69	0.18	3.2
372 Horse (Sarmatian)	-20.9	0.07	5.2	0.09	3.2
372 Horse (Sarmatian)	-20.6	0.07	3.74	0.14	3.2
378 Horse (Sarmatian)	-20.9	0.02	4.1	0.15	3.2
384 Cow (Alanic)	-20.4	0.13	6.21	0.17	3.2
384 Ovicaprid (Alanic)	-20.1	0.08	6.97	0.1	3.2
387 Ovicaprid (Sarmatian/Alanic)	-19.8	0.05	10.9	0.1	3.2
388 Horse (Sarmatian?)	-21.1	0.19	4.26	0.03	3.2
Ex1 Ch1 Horse	-20.5	0.01	5.95	0.28	3.3
Ex1S1 Ovicaprid	-20.3	0.05	6.81	0.04	3.2
Ex1S1 Ovicaprid	-19.9	0.01	7.66	0.02	3.2
<b>Koban settlement</b>					
Koban Sq 1 Bldg 1 Cow	-19.0	0.1	7.8	0.0	3.3
Koban Sq 1 Bldg 1 Ovicaprid	-19.7	0.1	6.5	0.2	3.4
Koban Sq 1 Bldg 1 Cow	-18.8	0.2	7.3	0.0	3.3
Koban Sq 1 Bldg 1 Pig	-17.8	0.1	8.1	0.2	3.3
Koban Sq 1 Bldg 1 Ovicaprid	-19.2	0.2	4.8	0.0	3.3

The Klin-Yar  $\delta^{15}\text{N}$  human data is non-normally distributed, but for the Sarmatian and Alanic groups, the bulk of the measurements cluster between 10–11‰. For the limited faunal samples available, cows yielded values around 6‰ and sheep around 7‰ (although there is one that is significantly higher<sup>7</sup>). A trophic level effect of ~4‰ is therefore apparent from these data, which is slightly less than the values identified by colleagues (O’Connell et al. 2003; Privat 2004). This might be taken to indicate a lack of obvious evidence for fish in the diet, but a significant complication is the absence of data regarding fish  $\delta^{15}\text{N}$  values from the region of the site. If freshwater fish  $\delta^{15}\text{N}$  values are enriched compared with the faunal values reported here, then it becomes less likely that fish is being consumed. If, on the other hand, fish  $\delta^{15}\text{N}$  values are little higher than terrestrial faunal values, then they could be consumed by humans in low numbers with the effect being masked. As mentioned already, fish remains are absent archaeologically at Klin-Yar and remain so until soil residue or food

<sup>7</sup>A hint regarding the possibility of a more wide-ranging, horse-based pastoralist economy during Sarmatian-Alanic times in which herds of stock were grazed over wider distances is provided by the range in ovicaprid  $\delta^{15}\text{N}$  values, which cover over 4‰. Iacumin et al. (2004) published data from Abganerovo, an archaeological site ~800 km north of Klin-Yar: they suggested that variation in archaeological sheep  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values could have been caused by the seasonal movement of herds, possibly to the Caspian Sea environs where plants typically include halophytes found in saline environments that have high  $\delta^{15}\text{N}$  values. Alternative explanations for the increased  $\delta^{15}\text{N}$  value of some animals may be related to penning and the increased nitrification derived from animal feces and manure (Hedges et al. 2005; Comisso and Nelson 2006; Bogaard et al. 2007). Again, unfortunately, we lack baseline isotopic data showing the variability in plant and animal values in the wider region in this period.

residue data is collected from other grave contexts at the site. This will necessitate new excavations, however.

The horse isotope values (all from Sarmatian and Alanic contexts) disclose some differences from the other faunal samples since they are depleted in both  $\delta^{15}\text{N}$  and  $\delta^{13}\text{C}$ . This may reflect the fact that they consumed slightly different types of plants and grasses compared with the other animals whose bones we have analyzed. Horses are often more depleted than ruminants in  $\delta^{15}\text{N}$  (Stevens and Hedges 2004). The values indicate that horses were clearly not a substantial part of the diet of these people, as indeed one might expect, because their  $\delta^{15}\text{N}$  values are less enriched than would be required given the trophic level effect.

Taken together, the stable isotope data are indicative of a mixed diet incorporating a  $\text{C}_4$  component during the Koban phase of occupation at Klin-Yar, shifting during the course of occupation by later Sarmatian and Alanic groups to a diet more dominated by resources derived via a  $\text{C}_3$  pathway. Our contention is that during these latter phases, freshwater fish were also incorporated into the dietary regime, resulting in  $^{14}\text{C}$  offsets, as shown in the nearby river using modern substrates. It is not known yet whether the same conclusion of fish exploitation can be extended to the Koban human population, in the absence of evidence for a reservoir effect within those burials.

## CONCLUSIONS

$^{14}\text{C}$  dating of human bone at the Klin-Yar site in the Russian North Caucasus has shown significant offsets from the ages expected based on historically dated artifacts associated with some individual inhumations. The  $^{14}\text{C}$  dates from humans are often older than their expected age. A correction to account for this offset is not applicable, because there is no correlation between the stable isotopes and the size of the offset. The most likely source for the old carbon affecting the  $^{14}\text{C}$  ages is the freshwater ecosystem, as demonstrated by the apparent ages obtained for dated flora and fauna collected live in 2005. No archaeological evidence for fish consumption has been found at the site. Excavations focused predominantly on the cemetery areas, however, so limited subsistence evidence associated with Sarmatian and Alanic settlement phases has been recovered. Somewhat controversially, in other Eurasian sites, isotopic evidence of freshwater fish consumption (up to 50% in the dietary protein) has been identified on the basis of isotopic evidence without any freshwater fish remains being discovered archaeologically (e.g. O'Connell et al. 2003), but the trophic level shifts are marked. In other contexts (e.g. NW Caspian), there is clearer evidence for probable fish consumption, with extremely large offsets present of up to 600 yr (Shishlina et al. 2007). In the present study, human bone collagen  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values did not provide clear evidence for fish consumption.

The implication for  $^{14}\text{C}$  dating human bone from these types of environments is quite clear; it must be undertaken with a high degree of caution. Stable isotope analysis of single amino acids and the application of  $\delta^{34}\text{S}$  (Leach et al. 2001; Privat et al. 2007) analysis to augment the routine use of carbon and nitrogen isotopes may help to shed further light on dietary pathways in humans and to avoid these types of errors. Similarly, for reliable chronology building, the most effective avenues are likely to be the dating of animal, rather than human, bone, and the dating of single amino acids selected to ensure that essential amino acids derived from organisms inhabiting non-atmospheric equilibrated reservoirs are avoided (McCullagh et al. 2006). It is to this end that we are now working.

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