

## FACTORS INFLUENCING THE RADIOCARBON DATING OF HUMAN SKELETAL REMAINS FROM THE DNIEPER RIVER SYSTEM: ARCHAEOLOGICAL AND STABLE ISOTOPE EVIDENCE OF DIET FROM THE EPIPALEOLITHIC TO ENEOLITHIC PERIODS

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**ABSTRACT.** Recent research has identified the existence of a freshwater reservoir effect influencing the radiocarbon dating of human skeletal remains from the Dnieper region of Ukraine (Lillie et al. 2009). The current study outlines the evidence for freshwater resource exploitation throughout the period ~10,200–3700 cal BC, and presents the available evidence for the existence of dietary offsets in the <sup>14</sup>C dates obtained. We have obtained human skeletal material from 54 Epipaleolithic to Mesolithic period individuals and 267 Neolithic to Eneolithic individuals, from 13 cemeteries, since our research in Ukraine began in 1992. Here, we present the initial results of stable isotope analysis of Eneolithic individuals from the Igren VIII cemetery alongside the Epipaleolithic to Eneolithic samples that have previously been analyzed. When contrasted against the evidence from the prehistoric fauna and fish remains studied, and modern fish species from the Dnieper region, we continue to see variability in diets at the population level, both internally and across cemeteries. We also observed temporal variability in human diets across these chronological periods. The fish samples (both archaeological and modern) show a wide range of isotope ratios for both  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , which could prove significant when interpreting the dietary sources being exploited. This information directly informs the <sup>14</sup>C dating program as an inherent degree of complexity is introduced into the dating of individuals whose diets combine freshwater and terrestrial sources in differing quantities and at differing temporal and/or spatial scales (e.g. Bronk Ramsey et al. 2014).

**KEYWORDS:** freshwater reservoir effect, diet, cemeteries, Dnieper Rapids.

### INTRODUCTION AND BACKGROUND

The Dnieper Rapids region of Ukraine (Figure 1) is unusual in a European context, in that it has a concentration of cemeteries dating from the Epipaleolithic through to Eneolithic/Copper Age periods (~10,200–3700 cal BC). The cemeteries in this region are characterized, in part, by the presence of extended burials, albeit with crouched inhumations occurring in the earliest phase of the sequence at Vasilyevka III (Epipaleolithic) and also in the latest phase considered here, at Molyukhov Bugor (Eneolithic). Pottery is found in the collective stages of burial at these cemeteries, during the Neolithic and later periods, and fish and deer tooth pendants occur as grave goods during all periods, with boar tusk plates (sewn onto clothing) in evidence during the later Mesolithic and Neolithic periods. The cemeteries are often delineated as Mariupol-type cemetery sites during the Neolithic phases of use (Telegin and Filenko 1982; Telegin 1986; Telegin and Potekhina 1987), although reanalysis of the chronology of the Mariupol-type cemeteries has shown that some of these cemeteries are actually of later Mesolithic date (e.g. Vasilyevka II and Marievka) (Telegin et al. 2002, 2003).

Additionally, as has been noted elsewhere, these cemeteries are of some interest as they are identified as “true cemeteries” in the sense that they are set apart from any settlement sites – and indeed evidence for associated settlements is rare (Telegin and Potekhina 1987). A number of the Dnieper Rapids cemeteries may have also functioned as territorial markers or as locations that indicated ancestral rights of access to the rich resources of the Dnieper River and its tributaries (Brinch Petersen and Meiklejohn 1995; Lillie 2008; Meiklejohn et al. 2009).

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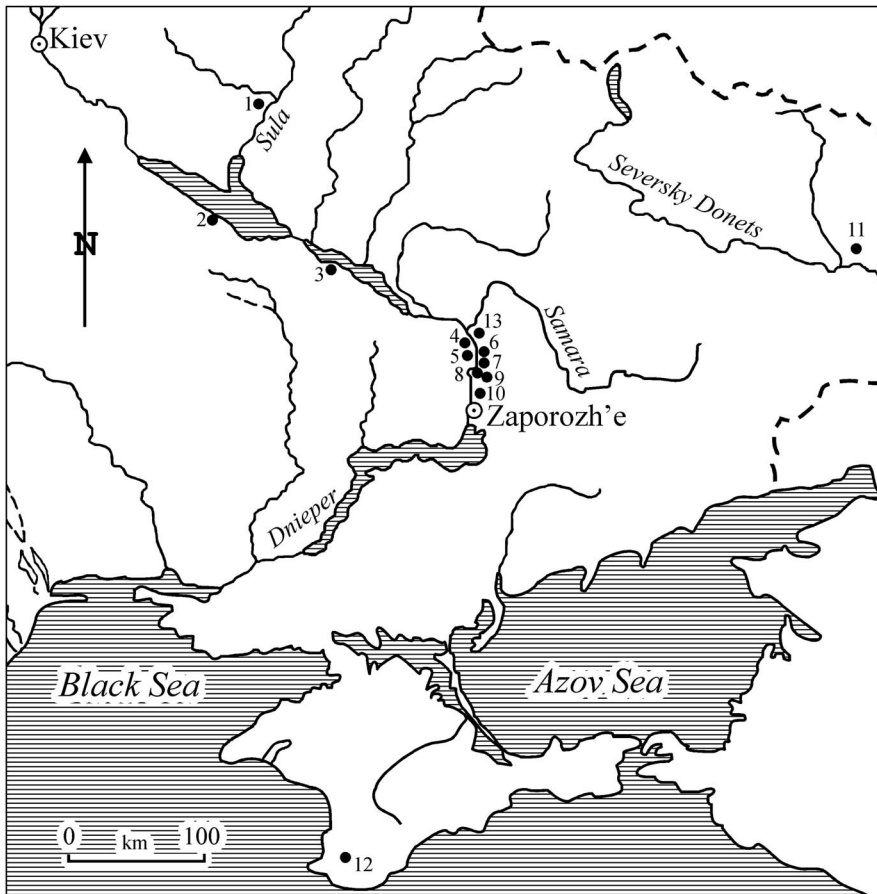


Figure 1 Key sites mentioned in the text. 1–Vyazivok, 2–Molukhov Bugor, 3–Dereivka I, 4–Voloskoe, 5–Nikolskoye, 6–Vasilyevka II, III and V, 7–Marievka, 8–Vovnigi I, 9–Yasinovatka, 10–Vil’nyanka, 11–Rogalik II, 12–Fat’Ma Koba, 13–Igren VIII.

From a subsistence standpoint, the isotope research in the Dnieper Rapids to date has focused on investigating the consumption of freshwater protein resources versus terrestrial protein resources, and the concomitant presence of a freshwater radiocarbon reservoir effect (Lillie and Jacobs 2006; Lillie et al. 2009). The continued consumption of high proportions of aquatic proteins from the Epipaleolithic through to the Neolithic period in the Dnieper Rapids region is evident from the human  $\delta^{15}\text{N}$  collagen values, which mainly demonstrate values of greater than 12‰ across the cemetery sites (Lillie and Richards 2000; Lillie et al. 2011). One of the main issues facing paleodietary reconstruction of prehistoric populations in the Dnieper Rapids region is the lack of significant quantities of associated fauna bone samples available for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  analysis, as these isotope values are used to provide an “isotopic baseline” for dietary interpretations (e.g. Schwarcz and Schoeninger 1991; Hedges and Reynard 2007). Previous  $^{14}\text{C}$  studies in the Dnieper Rapids regions, have identified evidence for a  $^{14}\text{C}$  freshwater reservoir effect at the sites of Dereivka 1 and Yasinovatka (with reservoir effects of ~250 yr and ~470 yr, respectively) (Lillie et al. 2009).

The core research questions central to understanding the diet and chronology of the study area are (i) to what proportion aquatic/freshwater protein contributes to prehistoric diets, (ii) does

the amount of aquatic protein consumed vary by chronological phase, and (iii) to what extent does the presence of a freshwater <sup>14</sup>C reservoir effect influence chronological interpretations of cemetery sites in the Dnieper Rapids region? These questions are addressed here by the integration of new carbon and nitrogen stable isotope analysis of sequential dentine samples from Igren VIII, alongside data sets of the existing stable isotope studies (Lillie and Richards 2000; Lillie et al. 2011).

**MATERIALS**

Overall, since the beginning of our isotopic research in 2000, we have studied 321 human samples, along with 26 fauna and 4 fish (archaeological) samples, from the associated cemeteries, with 8 modern fish samples included for comparison. In addition to the skeletal remains that have been analyzed to date, the current research further expands the study of prehistoric diets with the inclusion of dentine incremental analysis of 11 individuals from the Neolithic to Eneolithic phase of the cemetery population at Igren VIII (Table 1).

The site that forms the basis of this study is located in the Igren Peninsula on the left bank of the Samara River (Figure 1). Following on from earlier excavations in the 1930s and 1940s, D Ya Telegin and O S Filenko undertook five summer seasons of excavations between 1974 and 1978 (Telegin and Filenko 1982). During these excavations, 19 burials of Eneolithic date were excavated.

The skeletal remains interred in the cemetery of Igren VIII include two Neolithic(?) burials (Nos. 4 and 12) along with 17 Eneolithic Sredny Stog culture burials (numbers 1–3, 5–11, 13, 15–18, 26, 29) and two Yamna (Yamnaya in Russian) Culture (numbers 14, 20) burials (Telegin and Filenko 1982). In 12 of the graves, the bones were partially or completely “dusted” with ocher (1, 3, 5–8, 10, 13, 15, 16, 18, 29). Nine of the Sredny Stog burials (3, 5, 8, 10, 13, 15–18) contained burial goods, including flint, pottery, and jewelry. Burials 2, 5, and 10 had animal bone in association (the latter two examples are of beads from a possible headdress and bracelet, respectively). Associated finds also include a Trypillia stage B/C1 bowl and pot.

Table 1 Typological periodization and demographic data for the individuals analyzed (dentine increments) from the cemetery of Igren VIII. (skeleton numbers in column 2 are; 8 = Igren 8, 74–82 = dates skeletons were excavated i.e. 1974–1982, and n3a, 4, etc. skeleton numbers allocated during excavations).

Period	Skeleton number	Sex	Age
Neolithic?	8–74/4	Female	18–25 (~23)
Eneolithic	8–74/n3a	Male	young adult <23 yr
Eneolithic	8–74/n3a–320	Male	20–30 (probably around 25)
Eneolithic	8–74/3b	Female?	16
Eneolithic	8–74/8	Male	25–30
Eneolithic	8–76/13	Male	25–35 (probably ~30)
Eneolithic	8–76/16	Female	18–25 (younger end of range)
Eneolithic	8–78/19	Sub-adult	7 (5–7 range)
Eneolithic	8–78/23	Male	35–45 (~35–40)
Eneolithic	8–78/24	Female?	30–40 (~30–35)
Eneolithic	8–78/28	Male	40–50
Eneolithic	8–82/18	Indet. adult	20–30 (~25 max)

In the current study, 11 individuals from Igren VIII have been analyzed using high-resolution, incremental isotope analysis of the dentine in order to assess the relative contribution of dietary proteins. The individuals are coded with the site number, i.e. 8 for Igren VIII; the year of excavations, e.g. 74 for 1974; and the number allocated to the interment, such as n3a and n3a320 for the paired burial of a young adult male aged <23 yr and a male aged 20–30 yr (who was probably in the middle of this range) (Henderson 2015).

## METHODS

The methods used in the current study follow those outlined in Henderson et al. (2014). Due to the limitations of the skeletal archive, teeth were chosen on the basis of availability, with the pre-molars forming the majority of available teeth (the exception being individual 8–78/19 where the first maxillary molar was used). Only those teeth that displayed limited attrition, no caries, and no damage were selected. Demographic data is presented in Table 1. Each molar was cleaned using aluminium oxide air abrasive to remove adhering dirt. The tooth was halved using a Buehler Isomet with diamond-tipped blade. Collagen was extracted from one half of the tooth by demineralizing it in 0.5M HCl. Thereafter, the demineralized, but still intact, dentine was cut into 10 strips horizontally from the tip of the crown to the root apex using a scalpel, following Beaumont et al. (2012). The method used in this study cuts through the Andresen lines, making horizontal sections down a tooth from crown to root. In the crown of the tooth, the angle of the lines is roughly followed, although due to the size of the samples required each section represents many of these histological features. Toward the root there is an increased deviation from the histological features as they become more angled, meaning the samples are more time averaged. Each strip of demineralized dentine was rinsed in distilled water three times and heated at 70°C in pH3 H<sub>2</sub>O for 2 days, following a modified Longin (1971) method.

Following filtration and freeze-drying, the resulting collagen was weighed into tin capsules. Samples were measured in duplicate, at Oxford (ORAU, England), using a continuous-flow mass spectrometer (Sercon 20–22). A laboratory standard, alanine, was used to monitor data quality, check for drift and to calculate C:N ratios. The results were calibrated using a two-point calibration based on multiple aliquots of international standards, IAEACH6 and USGS40, which were included in the sample runs as well as multiple aliquots of the internal standard. Analytical reproducibility is  $\pm 0.1\%$  and  $\pm 0.2\%$  for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , respectively, based on multiple replicates of the internal laboratory standard.

The collagen samples obtained from each tooth strip were allocated an age range based on a developmental chart (Massler et al. 1941). The chart serves as a general indication of timing, although it is recognized that these ranges are approximate and that they are complicated by differences in development rates between individuals and populations. There is some evidence for differences in dentine development between males and females, with females showing slightly faster formation than males after the age of approximately 5 or 6 yr (Demirjian and Levesque 1980). As the sections made are approximate time intervals, no attempt at adjustment has been undertaken.

## RESULTS

In three instances, collagen was not be recovered from the processed sample; however, this does mean that 85 samples (increments) produced a suitable collagen yield for use in the current analysis. In all of these cases, the C:N ratios were considered acceptable at between 3.1 and 3.4 (DeNiro 1985). The possible Neolithic female from Igren VIII (Individual 8–74/4 [8744 in Figures 2 and 3]) has adult values of  $-18.6\%$  for  $\delta^{13}\text{C}$  and  $13.4\%$  for  $\delta^{15}\text{N}$  (unpublished data).

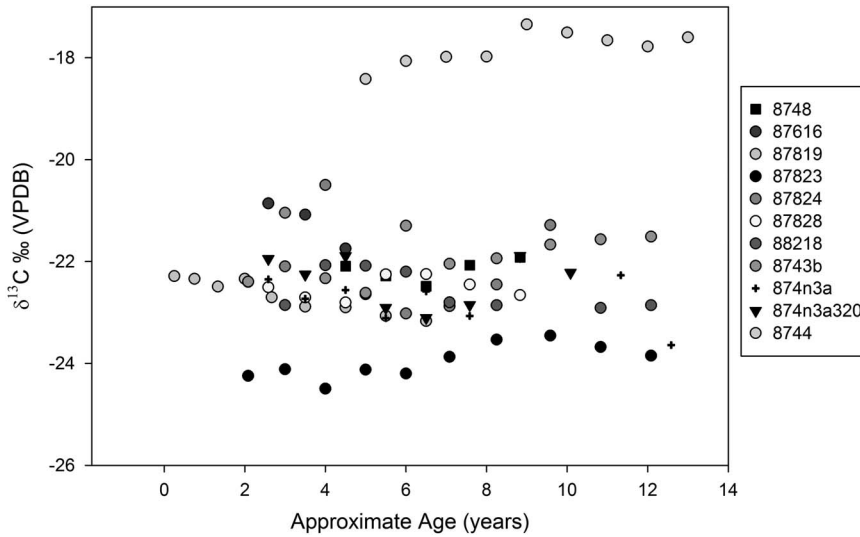


Figure 2 Childhood  $\delta^{13}\text{C}$  ratios for individuals from the cemetery of Igron VIII (Henderson 2015).

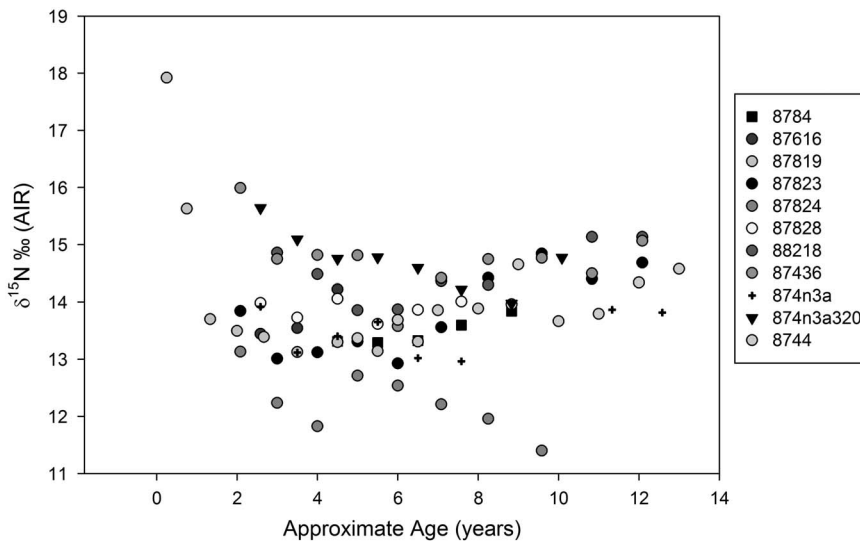


Figure 3 Childhood  $\delta^{15}\text{N}$  ratios for individuals from the cemetery of Igron VIII (Henderson 2015).

The preliminary dental  $\delta^{13}\text{C}$  ratios for this individual across the 4–9 yr age brackets in childhood indicate that this individual consumed a diet with  $\delta^{13}\text{C}$  ratios around  $-18.0\text{‰}$ , while as can be seen in Figure 2, the  $\delta^{15}\text{N}$  ratios across this period of childhood are between  $\sim 13$  to  $14\text{‰}$  (Figure 3).

For the Eneolithic period, all of the individuals studied exhibit  $\delta^{13}\text{C}$  ratios between ca.  $-24.0\text{‰}$  and  $-21.0\text{‰}$  ( $-22.6 \pm 0.8\text{‰}$ ) from birth through to  $\sim 14$  yr of age (Figure 2). By contrast, there is more variation in evidence in the  $\delta^{15}\text{N}$  ratios for this period (Figure 3). Individual 8–78/19

(87819), a child aged 7 yr at death, has a noticeably higher  $\delta^{15}\text{N}$  ratio of  $\sim 18\text{‰}$  at 6 months, lowering to  $\sim 13.3\text{‰}$  at 2.5 yr. While the data for individual 8-74/3b (8743b), a  $\sim 16$ -yr-old female(?), begins at 2 yr of age, the data again show a reduction from more elevated  $\delta^{15}\text{N}$  values (starting at  $16.0\text{‰}$ ), which appear to stabilize at around 2.5 yr of age at  $14.7\text{‰}$ .

While the isotope data are less clear for individuals 8-74/n3a-320 (male  $\sim 25$  yr of age at death) and 8-78/23 (male  $\sim 35$ – $40$  at death), it would appear that the initially higher nitrogen ratios are again stabilizing at  $\sim 2.5$ – $3$  yr of age for these individuals. As can be seen in Figure 3, for all of the individuals studied a relatively consistent nitrogen stable isotope signature is achieved by  $\sim 2$ – $3$  yr of age, after the initial higher levels in early childhood, and that once “stabilized” these ratios remain relatively constant throughout the biological age range of 2–14 yr.

The only outlier values for  $\delta^{15}\text{N}$  occur in relation to individual 8-78/24, a female(?) aged  $\sim 30$ – $35$  yr at death. Between 2–4 yr of age, the  $\delta^{15}\text{N}$  values drop from to  $13.1\text{‰}$  to  $11.8\text{‰}$  subsequently rising to  $12.7\text{‰}$  at 4.5 yr and then decreasing steadily to  $11.4\text{‰}$  by 10 yr of age. Admittedly, the overall variation in  $\delta^{15}\text{N}$  for this individual is only  $1.7\text{‰}$ , but it is the overall decreasing chronological trend in  $\delta^{15}\text{N}$  values that makes this individual stand out from the group. Interestingly, the  $\delta^{13}\text{C}$  values for this individual exhibit the opposite trend in that they shift from  $-22.4\text{‰}$  to  $-20.5\text{‰}$  between 2.5 and 4 yr of age, reducing to a minimum of  $-23.0\text{‰}$  at 6 yr of age, and subsequently rising steadily to  $-21.3\text{‰}$  by  $\sim 9.5$  yr of age. Unfortunately, at this stage in our analyses, justification for these trends is not readily apparent.

Finally, individual 8-78/23 (Male aged  $\sim 35$ – $40$ ) has the most negative  $\delta^{13}\text{C}$  ratios recorded for this population, with values of  $\sim -24.0\text{‰}$  recorded across the period 2–12 yr of age. The associated  $\delta^{15}\text{N}$  values elevate across the period 3–12 yr of age from  $13.0\text{‰}$  to  $14.7\text{‰}$ .

### Interpretation of the Igren VIII Data

The dental stable isotope analysis of the individuals from the Igren VIII cemetery indicate that in early childhood, the elevated nitrogen ratios in evidence are associated with the infant diet prior to weaning. In addition, the data suggest that in this particular population the children are weaned by  $\sim 2$ – $3$  yr of age. The reasons behind age of weaning in prehistoric populations have been shown to be complex (e.g. Sellen and Smay 2001), but the reliability of the resource base and nutritional value of the post-weaning diet clearly contribute to the weaning ages in the Dnieper groups. Lillie (2008) has previously noted that a study of the frequency of enamel hypoplasia's on the dentitions of the Dnieper Rapids populations has shown that very low levels of stress occur between the ages of 2.0–6.0 yr of age, across the Epipaleolithic to Neolithic periods. As noted elsewhere, however (e.g. Goodman et al. 1984), hypoplasias are nonspecific childhood stress indicators, and as such they do not necessarily reflect the occurrence of a weaning event. As such, the  $\delta^{15}\text{N}$  ratios presented in Figure 3 may provide a more robust method for assessing the timing of this event in the prehistoric populations of the Dnieper Rapids region of Ukraine.

Post-weaning, the data appear to be relatively consistent across the childhood years of the Eneolithic individuals, with the obvious exception of individual 8-78/24 (discussed below), such that by 12 yr of age all of the individuals studied are clearly consuming diets in which freshwater resources, such as fish and mollusks, are making up a significant proportion of the dietary protein, and the data also indicate that that freshwater resources made up a significant proportion of childhood diets, from  $\sim 2$ – $3$  yr through to  $\sim 14$  yr of age. Importantly, this is the first time that we have obtained data on the composition of childhood diets from the Dnieper Rapids cemeteries of Ukraine.



Table 2 AMS dates on fish, human and fauna samples from the cemeteries of Dereivka 1 and Yasinovatka (Lillie et al. 2011: Table 1).

Cemetery	Sample	Element	Age (yr)	Lab nr	Radiocarbon age (uncal BP)	$\delta^{13}\text{C}$ (‰)	$\delta^{15}\text{N}$ (‰)
Dereivka	Fish (indet.)	Teeth	Mature	OxA-X-2222-33	6915 ± 50	-28.0	8.3
	Human (ind. 29)	Skull	40–50	OxA-17495	6398 ± 35	-22.5	12.7
	Red deer	Antler	Mature	OxA-17496	6147 ± 35	-21.3	7.7
Yasinovatka	Fish	Teeth	Mature	OxA-17498	6840 ± 37	-16.2	13.9
	Human (ind.54)	Skull	30–40	OxA-17500	6593 ± 35	-22.1	14.1
	Red deer	Teeth	Mature	OxA-17499	6121 ± 34	-18.8	6.8

## DISCUSSION: THE WIDER CONTEXT OF THE DNEIPER RIVER SYSTEM

There is now considerable stable isotope data to support the consumption of freshwater resources by the prehistoric populations of the Dnieper Rapids region across the period 10,000–3000 cal BC (Lillie et al. 2003, 2009, 2011, this paper). This data reinforces the archaeological evidence for fishing and shellfish gathering in the Dnieper catchment (e.g. Telegin 1986; Telegin and Potekhina 1987), and the addition of Igren VIII to our studies of diet for the populations occupying the Middle and Lower Dnieper Basin cemeteries and campsites across the Epipaleolithic–Neolithic/Copper Age further enhances our understanding of the nature and duration of the consumption of these resources (i.e. from early childhood in the case of Igren VIII).

To date, the sites of Vasilyevka III, Dereivka I and II, Vil'nyanka, Yasinovatka, Nikolskoye and Molukhov Bugor, along with Voloshkoe, Vasilyevka II, Marievka, Fat'ma Koba (Crimea), Vyazivok, Rogalik II, and Vovnigi I have been analyzed using either AMS dating or a combination of AMS dating and stable isotopes analyses (Lillie and Richards 2000; Lillie and Jacobs 2006; Lillie et al. 2009, 2011).

While the sites and cemeteries of Vasilyevka III, Vyshgorod, Dobryanka, Vasilyevka II, Igren VIII (Mesolithic), Vil'nyanka, Osipovka, Dereivka I, Yasinovatka, and Nikolskoye have all been AMS dated, it is perhaps now recognized that the more significant of these sites, from a dating perspective, are the Neolithic cemeteries of Dereivka 1 and Yasinovatka, where the analysis of a combination of (closely associated) human, faunal, and fish remains has shown that there are significant offsets in the  $^{14}\text{C}$  determinations between samples from the same grave context (Table 2). The fish and deer teeth are recovered from necklaces, pendants, or bracelets that were worn by the deceased when they were buried (Telegin and Potekhina 1987).<sup>1</sup>

<sup>1</sup>Unfortunately, the available material (both in terms of sample size and elements for use in analysis) did not permit the stable isotope analysis of the fish and deer tooth pendants from all of the burials in the Dnieper Rapids, as not all associated material culture artifacts were curated, and even where samples were curated only limited access for sampling was permitted (Lillie et al. 2011: Table 1). Quoted  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values for fish samples were obtained during sample preparations for AMS dating at the ORAU; as such, these values should not be used in interpretations of dietary isotope data due to differences in the processing protocols between ratios obtained for dietary studies and those obtained for AMS dating purposes.

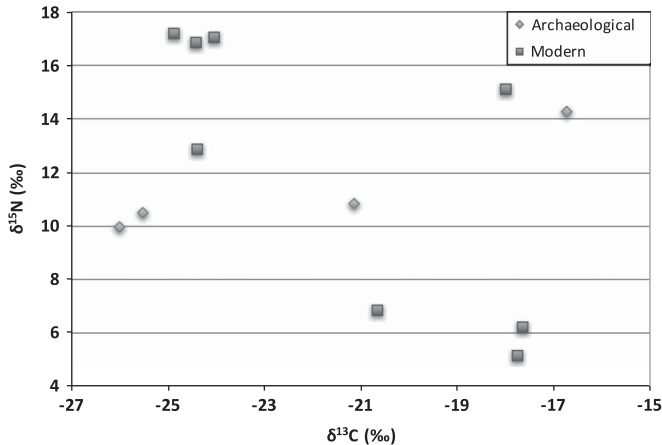


Figure 4 Archaeological and modern fish stable isotope values from the Dnieper River system. Archaeological samples are carp and pearl roach. Modern samples from the Dnieper and Chernovtsky rivers include silver carp, gray pike, European perch, common carp, Crucian carp, and silver carp (Budd 2007).

The differences (offsets) observed between the  $^{14}\text{C}$  determinations in Table 2 are clearly due to the different dietary sources exploited by humans, combined with the occurrence of older carbon in the fish samples (carp or pearl roach) (Lillie et al. 2011). In addition, as noted elsewhere (e.g. Lake Baikal; Bronk Ramsey et al. 2014), the significant variability in  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values evidenced by the fish remains from the Dnieper (both archaeological and modern samples) (Figure 4) indicates that  $\delta^{13}\text{C}$  ratios are difficult to correlate to  $^{14}\text{C}$  offsets (Budd 2007; Bronk Ramsey et al. 2014: 791). The  $\delta^{13}\text{C}$  isotope ratios for the human samples at Dereivka 1 (male 40–50, cortical bone of skull) and Yasinovatka (male 30–40 cortical bone of skull) are  $-22.4\text{‰}$  and  $-22.6\text{‰}$ , respectively. In isolation, these values would potentially suggest the consumption of  $\text{C}_3$  terrestrial proteins, given the values obtained from terrestrial fauna in this region (Lillie et al. 2009, 2011), but in combination with the elevated  $\delta^{15}\text{N}$  ratios exhibited by these individuals and the archaeological  $\delta^{15}\text{N}$  values of fish from the Dnieper (Figure 4), the values clearly demonstrate the contribution of freshwater proteins into the diet (see also Wood et al. 2013).

The  $\delta^{15}\text{N}$  ratios of  $12.7\text{‰}$  at Dereivka 1 (male 40–50, cortical bone of skull) and  $14.0\text{‰}$  at Yasinovatka (male 30–40 cortical bone of skull) (Table 2) would potentially indicate that individual 54 at Yasinovatka is consuming proportionally higher quantities of freshwater protein than terrestrial protein when compared to individual 29 at Dereivka 1. However, it is recognized that considerable variability occurs within freshwater systems (Philippsen 2013; Fernandes et al. 2014), and these two cemeteries are some distance apart within the Dnieper system. Despite this, it is apparent that these differences in diet correlate with a larger offset from the terrestrial baseline at Yasinovatka (472 yr) than the offset in evidence at Dereivka I (251 yr). While linear interpolation is not viable on the basis of this data, in this example a  $1.3\text{‰}$  elevation in  $\delta^{15}\text{N}$  at Yasinovatka (as compared to the Dereivka 1 sample) coincides with a greater (i.e. 470 yr) offset from the terrestrial baseline sample when contrasted with the  $12.7\text{‰}$   $\delta^{15}\text{N}$  ratio at Dereivka I (where a 250-yr offset is evident). It is anticipated that this variability will be investigated in greater detail in future studies as at face value the higher human  $\delta^{15}\text{N}$  value at Yasinovatka equates to a larger FRE offset from the terrestrial baseline, but this is



complicated by the differences in fish  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values between these two locations (Table 2) (Lillie et al. 2011).

Fauna and fish stable isotope values from burials within Yasinovatka and Dereivka indicate that the terrestrial fauna have isotope values of  $-20.0\text{‰}$  ( $\delta^{13}\text{C}$ ) and  $7.5\text{‰}$  ( $\delta^{15}\text{N}$ ) for a deer tooth (incisoform, I2) in burial 28 at Yasinovatka and  $-20.4\text{‰}$  ( $\delta^{13}\text{C}$ ) and  $4.9\text{‰}$  ( $\delta^{15}\text{N}$ ) for a deer antler sample from Dereivka (Lillie et al. 2011). The variability between these samples is likely a function of the two different elements analyzed, e.g. tooth dentine vs. antler collagen. Tooth dentine forms incrementally over a period of months (Mitchell and Youngson 1969; Brown and Chapman 1991; Sponheimer et al. 2003); therefore, the bulk dentine sample represents a mixed dietary signal, including periods of nursing (see Balasse et al. 2001 for comparative research on cattle dentine samples). Conversely, red deer antlers form at approximately 1 year of age (Clutton-Brock 1982), and so reflect a postweaning phase of the diet. Therefore, one would expect mature antler collagen to exhibit lower  $\delta^{15}\text{N}$  values than a bulk dentine sample.

The archaeological fish tooth from burial 56 at Yasinovatka (Figure 4 – center) has values of  $-21.1\text{‰}$  ( $\delta^{13}\text{C}$ ) and  $10.8\text{‰}$  ( $\delta^{15}\text{N}$ ), while at Dereivka the fish from burials 5 and 46 have values of  $-16.8\text{‰}$  and  $-25.5\text{‰}$  for  $\delta^{13}\text{C}$  and  $14.3\text{‰}$  and  $10.5\text{‰}$  for  $\delta^{15}\text{N}$ , respectively (Figure 4 – right and left sides of distribution) (Lillie et al. 2011:64–5). Along with the Neolithic sample from Vil'nyanka (Figure 4, far left), these values clearly indicate a significant potential for a wide range of offsets within a given cemetery population in the Dnieper Rapids region, depending on which sources of dietary foodstuffs are being exploited (see for e.g. Rouja et al. 2003). In addition, there is clearly an established potential for significant intercemetery variability both between individuals and chronological periods.

To date, stable isotope values have been recorded for the Epipaleolithic cemetery of Vasilyevka III, where Lillie et al. (2003) analyzed 21 individuals (both males and females), all of whom had  $\delta^{15}\text{N}$  values over  $11.9\text{‰}$  (average =  $12.7 \pm 0.6\text{‰}$ ), with a maximum value of  $14.1\text{‰}$  recorded on an adult male aged  $>55$  yr, and  $\delta^{13}\text{C}$  values between  $-22.5\text{‰}$  and  $-21.6\text{‰}$  (average =  $-22.2 \pm 1.5\text{‰}$ ). Faunal samples obtained during recent analyses have shown that at Mesolithic sites in the region (e.g. Vyazivok, Igren VIII, and Rogalik 2) and further south in Crimea,  $\delta^{15}\text{N}$  values of  $5.5\text{‰}$  (*Cervus elaphus*) through to  $8.8\text{‰}$  (*Sus scrofa*) are evidenced (Lillie et al. 2011). As such, the human isotope data are interpreted as indicating a relatively uniform diet, with a strong dependence on freshwater and terrestrial animal protein.

Towards the latter part of the Mesolithic period, the cemetery sites of Vasilyevka II and Marievka have produced contrasting indicators of diet, with Marievka (7000–6200 cal BC) having an individual (burial 10, male, 35–45 yr) dated to 7020–6100 cal BC at  $2\sigma$  (OxA-6200,  $7620 \pm 160$  BP) with a  $\delta^{13}\text{C}$  ratio of  $-21.7\text{‰}$  and a  $\delta^{15}\text{N}$  ratio of  $13.0\text{‰}$ , while two other individuals (burials 4 [male? 50–60 yr]) dated to 7045–6690 cal BC (OxA-6199,  $7955 \pm 55$  BP) and 14 [indet. adult aged 35–45], dated to 6685–6240 cal BC (OxA-6269,  $7630 \pm 110$  BP), dated to 6685–6240 cal BC (OxA-6269,  $7630 \pm 110$  BP) had  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values of  $-22.0\text{‰}$  and  $-22.1\text{‰}$  and  $10.1\text{‰}$  and  $10.8\text{‰}$ , respectively (all dates and isotope values were obtained from cortical bone from the skull of each individual) (Lillie et al. 2011). Given the available fish and fauna values, it is clear that individuals 4 and 14 (adult male/adult of indeterminate sex) were consuming diets that consisted principally of terrestrial resources, while individual 10 (adult male) was consuming a diet with a stronger dependence on freshwater resources (Lillie et al. 2011:61–4).

At the later Mesolithic site of Vasilyevka II, dated to  $\sim 7300$ – $6220$  cal BC, analysis of 14 individuals by Lillie and Jacobs (2006) led to the observation that both males and females

consumed diets that were rich in freshwater resources, with  $\delta^{13}\text{C}$  values ranging from  $-21.8$  to  $-20.1\text{‰}$  ( $-20.9 \pm 0.5\text{‰}$ ) and  $\delta^{15}\text{N}$  ratios ranging from  $12.4$  to  $14.7\text{‰}$  ( $13.4 \pm 0.7\text{‰}$ ). These authors also concluded that when compared to other individuals of Mesolithic age from the cemeteries of Marievka (discussed above), Dereivka I, and Osipovka, the Vasilyevka II populations were generally placing a greater emphasis on the exploitation of freshwater resources alongside terrestrial protein sources, although variability is again in evidence (e.g. individual 10 at Marievka).

For the Neolithic period, the cemeteries of Vovnigi I, Yasinovatka, Dereivka I, Vil'nyanka, Nikolskoye, and the site of Molukhov Bugor (a total of 43 human samples, 13 fauna, and 4 fish samples) have all been studied (Lillie et al. 2011). The fauna include a deer antler from Dereivka I that produced a  $\delta^{13}\text{C}$  value of  $-20.4\text{‰}$  and a  $\delta^{15}\text{N}$  ratio of  $4.9\text{‰}$ , while a deer tooth sample from Yasinovatka has produced ratios of  $-20.0\text{‰}$  and  $7.4\text{‰}$  for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , respectively, and a deer tooth sample from Vovnigi I produced ratios of  $-19.1\text{‰}$  and  $6.9\text{‰}$  for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , respectively. The Neolithic fauna from Molukhov Bugor produced ranges between  $-23.2\text{‰}$  (*Cervus elaphus*) to  $-18.9\text{‰}$  (*Ursus arctos*) for  $\delta^{13}\text{C}$  and  $4.8\text{‰}$  (*Sus scrofa*) to  $7\text{‰}$  (*Ursus arctos*) for  $\delta^{15}\text{N}$ . Intermediate values were recorded for additional pig, cattle, and sheep/goat samples from this site.

The human stable isotope values from Nikolskoye cluster between  $-23.6\text{‰}$  and  $-19.6\text{‰}$  for  $\delta^{13}\text{C}$  ( $-22.7 \pm 1.2\text{‰}$ ) and  $12.2\text{‰}$  to  $14.3\text{‰}$  for  $\delta^{15}\text{N}$  ( $13.4 \pm 0.7\text{‰}$ ). These ratios are clearly elevated when compared to the contemporary fauna, and the situation is the same at Yasinovatka where values of  $-24.1\text{‰}$  to  $-22.4\text{‰}$  (average =  $-23.4 \pm 0.6\text{‰}$ ) and  $11.4\text{‰}$  to  $15.1\text{‰}$  (average =  $13.6 \pm 1.1\text{‰}$ ) are recorded for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , respectively. At Dereivka I, values of  $-23.6\text{‰}$  to  $-21.7\text{‰}$  (average =  $-23.1 \pm 0.7\text{‰}$ ) and  $10.0\text{‰}$  to  $13.5\text{‰}$  ( $11.6 \pm 1.3\text{‰}$ ) are recorded for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ , respectively. The values for the humans are difficult to interpret due to the variable faunal ranges for the period, although it is apparent that freshwater resources (fish) are contributing some proportion of the dietary proteins at these sites. This is reinforced by the fish values from Dereivka I, Vil'nyanka, and Yasinovatka, which exhibit very wide ranges for  $\delta^{13}\text{C}$ , between  $-26\text{‰}$  and  $-16.8\text{‰}$  and  $\delta^{15}\text{N}$  ratios of  $10\text{‰}$  to  $14.3\text{‰}$ . While limited in number, the available human bone samples from Vasilyevka V have produced dietary isotope compositions with  $\delta^{13}\text{C}$  ranging between  $-23.2\text{‰}$  and  $-20.2\text{‰}$  and  $\delta^{15}\text{N}$  between  $10.0\text{‰}$  and  $12.3\text{‰}$ , again the isotope values of certain individuals are indicative of the consumption of freshwater resources. Interestingly, while the two  $\delta^{15}\text{N}$  ratios of  $10.0\text{‰}$  and  $10.6\text{‰}$  at Vasilyevka V might suggest that these individuals had a mainly terrestrial resource oriented diet, the  $\delta^{13}\text{C}$  values of  $-22.4\text{‰}$  and  $-23.2\text{‰}$  would lend weight to the observation that freshwater resources were being consumed. The single individual with an elevated  $\delta^{15}\text{N}$  ratio of  $12.3\text{‰}$  probably consumed a diet with a higher input of freshwater resources, and the  $\delta^{13}\text{C}$  ratio of  $-22.3\text{‰}$  for this individual reinforces this observation (Lillie et al. 2011:65).

During the Eneolithic period at the site of Molukhov Bugor, which is located higher up in the Dnieper River catchment, the stable isotope data are broadly consistent with the Neolithic data, although only four human samples were available for analysis from this location. The human samples range between  $-23.1\text{‰}$  and  $-21.2\text{‰}$  (average =  $-21.8 \pm 1\text{‰}$ ) for  $\delta^{13}\text{C}$  and  $11.5\text{‰}$  to  $12.9\text{‰}$  (average =  $12.2 \pm 0.7\text{‰}$ ) for  $\delta^{15}\text{N}$ . The Eneolithic faunal samples from Molukhov Bugor exhibit a range of  $-22.6\text{‰}$  to  $-19.8\text{‰}$  (average =  $-21.5 \pm 1.1\text{‰}$ ) for  $\delta^{13}\text{C}$  and  $5.6\text{‰}$  to  $7.5\text{‰}$  (average =  $6.7 \pm 0.7\text{‰}$ ) for  $\delta^{15}\text{N}$ . As such, it is also likely that freshwater resources, alongside terrestrial herbivores, contributed to the Eneolithic diet at Molukhov Bugor.

## THE PROBLEM WITH FISH

In summary, it is clear from the isotope data and archaeological record that the inclusion of fish protein in prehistoric diets was a major factor in the Dnieper Rapids region. Returning to the core study themes of this paper, it is evident from the  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values that (i) in most cases freshwater protein contributed a large (if not major) proportion of the diet for the prehistoric populations throughout the Epipaleolithic to Eneolithic periods. In addition, (ii) the amount of freshwater protein appears to be largely consistent throughout the chronological phases; the earliest dated site in the sequence, i.e. Vasilyevka III (at ~10,000 cal BC) has clear evidence for the consumption of freshwater protein (Lillie et al. 2003), and this continues through to the Eneolithic/Copper Age at Igren VIII, up to ~3000 cal BC, although the pattern for freshwater protein consumption exhibits variation at both the inter- and intrasite levels of analysis. Finally, the <sup>14</sup>C determinations of human, fauna, and fish collagen samples recovered in close association, demonstrate very convincing evidence for a freshwater reservoir effect, ranging between ~250 and 470 yr, dependent on the feeding habits of the species of fish consumed (benthic versus bottom feeders, etc.) and the proportion of fish protein consumed in an individual's diet.

A fundamental problem with this approach to paleodietary reconstruction lies in the fact that the fish isotope ratios are not consistent at the intra- and interspecies levels (fish can be deep or shallow water feeders, predators, and benthic feeders), and the ethnographic evidence for selectivity in human consumption patterns of fish would seem to negate any easy linear interpolation of the nitrogen ratios that are in evidence. The stable isotope data for individual 8-74/4 from Igren VIII (8744 in Figure 2) would suggest that, in light of the available fish ratios from the Dnieper Rapids (Figure 4), this individual could either have placed a very particular dietary focus on a single species, or was consuming a diet that differed markedly to the later (Eneolithic) individuals at this location. This individual appears anomalous in relation to the individuals from the Dnieper Rapids region during the prehistoric period, and recent dating of this individual has indicated that as opposed to being of Neolithic date, individual 4 from Igren VIII is in fact an intrusive interment.<sup>2</sup> While the consumption of freshwater resources occurs throughout the prehistoric period, we can postulate that the low  $\delta^{15}\text{N}$  values at Igren VIII (e.g. individual 8-78/24 [87824 in Figure 2]) might indicate that there are some human individuals with less potential for a reservoir offset. This could be assessed using associated faunal remains from burials, thus potentially offering a way to limit the biases that occur when dating these populations.

In conclusion, it is clear from the dating and isotope studies undertaken to date (e.g. Lillie et al. 2009, 2011) that the majority of dates on individuals with elevated nitrogen ratios (i.e. above ~12‰) should be considered to have a potential reservoir effect. As noted above, at ~12.7‰ at Dereivka this is a 250-yr offset from the terrestrial baseline (the issue of limited faunal values notwithstanding), while at 14.0‰ at Yasinovatka the offset rises to 470 yr. At this stage in the research agenda, it is clear that targeted excavations aimed at recovering a combination of human, fauna, and where possible, fish samples, are needed in order to adequately determine potential degrees of offset, and the actual offset in <sup>14</sup>C yr, alongside the stable isotope values for the populations studied (e.g. Schulting et al. 2014).

<sup>2</sup>Since the initial study was undertaken, AMS dating of individual 8-74/4 from Igren VIII has shown that this individual is in fact an intrusive burial of Medieval date, as opposed to the earlier Neolithic age suggested by Telegin and Zaliznyak (1975) and Telegin and Potekhina (1987) for seven interments at this location, and that this individual is most likely to be from a Slavic village that overlies the Eneolithic settlement of Igren VIII (Gorobets and Matlaev 2014; A Nikitin, personal communication, 2016). In addition to a combination of domestic and wild fauna species consumed at this Medieval settlement, fish and fowl (primarily ducks, including mallard) are attested, which would help account for the isotope ratios in evidence from individual 8-74/4.

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