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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 ¹⁴C 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 δ^{13} C and δ^{15} N, which could prove significant when interpreting the dietary sources being exploited. This information directly informs the ¹⁴C 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 Eurpean 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 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 δ^{15} 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 δ^{13} C and δ^{15} 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 ¹⁴C studies in the Dnieper Rapids regions, have identified evidence for a ¹⁴C 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 ¹⁴C 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.

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

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)

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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).

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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 H₂O 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 δ^{13} C and δ^{15} 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 114 a developmental chart (Massler et al. 1941). The chart serves as a general indication of timing, 115 although it is recognized that these ranges are approximate and that they are complicated by 116 differences in development rates between individuals and populations. There is some evidence 117 for differences in dentine development between males and females, with females showing 118 slightly faster formation than males after the age of approximately 5 or 6 yr (Demirjian and 119 Levesque 1980). As the sections made are approximate time intervals, no attempt at adjustment 120 has been undertaken. 121

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 δ^{13} C and 13.4% for δ^{15} N (unpublished data).



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



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

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

For the Eneolithic period, all of the individuals studied exhibit δ^{13} C ratios between ca. -24.0% ¹³¹ and -21.0% (-22.6 ± 0.8%) from birth through to ~14 yr of age (Figure 2). By contrast, there is ¹³² more variation in evidence in the δ^{15} N ratios for this period (Figure 3). Individual 8–78/19 ¹³³

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

While the isotope data are less clear for individuals 8-74/n3a-320 (male ~ 25 yr of age at death)137and 8-78/23 (male $\sim 35-40$ at death), it would appear that the initially higher nitrogen ratios are138again stabilizing at $\sim 2.5-3$ yr of age for these individuals. As can be seen in Figure 3, for all of139the individuals studied a relatively consistent nitrogen stable isotope signature is achieved by140 $\sim 2-3$ yr of age, after the initial higher levels in early childhood, and that once "stabilized" these141ratios remain relatively constant throughout the biological age range of 2-14 yr.142

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The only outlier values for δ^{15} N occur in relation to individual 8–78/24, a female(?) aged ~30–35 yr at death. Between 2–4 yr of age, the δ^{15} N values drop from to 13.1% to 11.8% subsequently rising to 12.7% at 4.5 yr and then decreasing steadily to 11.4% by 10 yr of age. Admittedly, the overall variation in δ^{15} N for this individual is only 1.7%, but it is the overall decreasing chronological trend in δ^{15} N values that makes this individual stand out from the group. Interestingly, the δ^{13} C values for this individual exhibit the opposite trend in that they shift from –22.4% to –20.5% between 2.5 and 4 yr of age, reducing to a minimum of –23.0% at 6 yr of age, and subsequently rising steadily to –21.3% by ~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 ~35–40) has the most negative δ^{13} C ratios recorded for this population, with values of ~-24.0% recorded across the period 2–12 yr of age. The associated δ^{15} N values elevate across the period 3–12 yr of age from 13.0% to 14.7%.

Interpretation of the Igren VIII Data

The dental stable isotope analysis of the individuals from the Igren VIII cemetery indicate that 156 in early childhood, the elevated nitrogen ratios in evidence are associated with the infant diet 157 prior to weaning. In addition, the data suggest that in this particular population the children 158 are weaned by $\sim 2-3$ yr of age. The reasons behind age of weaning in prehistoric populations 159 have been shown to be complex (e.g. Sellen and Smay 2001), but the reliability of the resource 160 base and nutritional value of the post-weaning diet clearly contribute to the weaning ages in the 161 Dnieper groups. Lillie (2008) has previously noted that a study of the frequency of enamel 162 hypoplasia's on the dentitions of the Dnieper Rapids populations has shown that very low levels 163 of stress occur between the ages of 2.0–6.0 yr of age, across the Epipaleolithic to Neolithic 164 periods. As noted elsewhere, however (e.g. Goodman et al. 1984), hypoplasias are nonspecific 165 childhood stress indicators, and as such they do not necessarily reflect the occurrence 166 of a weaning event. As such, the δ^{15} N ratios presented in Figure 3 may provide a more robust 167 method for assessing the timing of this event in the prehistoric populations of the Dnieper 168 Rapids region of Ukraine. 169

Post-weaning, the data appear to be relatively consistent across the childhood years of the Eneo-170lithic individuals, with the obvious exception of individual 8–78/24 (discussed below), such that by17112 yr of age all of the individuals studied are clearly consuming diets in which freshwater resources,172such as fish and mollusks, are making up a significant proportion of the dietary protein, and the173data also indicate that that freshwater resources made up a significant proportion of childhood174diets, from ~2–3 yr through to ~14 yr of age. Importantly, this is the first time that we have obtained175data on the composition of childhood diets from the Dnieper Rapids cemeteries of Ukraine.176

Cemetery	Sample	Element	Age (yr)	Lab nr	Radiocarbon age (uncal BP)	δ ¹³ C (‰)	$\frac{\delta^{15}N}{(\%)}$
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

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

DISCUSSION: THE WIDER CONTEXT OF THE DNIEPER RIVER SYSTEM

There is now considerable stable isotope data to support the consumption of freshwater 177 resources by the prehistoric populations of the Dnieper Rapids region across the period 178 10,000-3000 cal BC (Lillie et al. 2003, 2009, 2011, this paper). This data reinforces the 179 archaeological evidence for fishing and shellfish gathering in the Dnieper catchment 180 (e.g. Telegin 1986; Telegin and Potekhina 1987), and the addition of Igren VIII to our studies of 181 diet for the populations occupying the Middle and Lower Dnieper Basin cemeteries 182 and campsites across the Epipaleolithic-Eneolithic/Copper Age further enhances our 183 understanding of the nature and duration of the consumption of these resources (i.e. from 184 early childhood in the case of Igren VIII). 185

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).

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While the sites and cemeteries of Vasilyevka III, Vyshgorod, Dobryanka, Vasilyevka II, 191 Igren VIII (Mesolithic), Vil'nyanka, Osipovka, Dereivka I, Yasinovatka, and Nikolskoye 192 have all been AMS dated, it is perhaps now recognized that the more significant of these 193 sites, from a dating perspective, are the Neolithic cemeteries of Dereivka 1 and Yasinovatka, 194 where the analysis of a combination of (closely associated) human, faunal, and fish remains 195 has shown that there are significant offsets in the ¹⁴C determinations between samples from 196 the same grave context (Table 2). The fish and deer teeth are recovered from necklaces. 197 pendants, or bracelets that were worn by the deceased when they were buried (Telegin and 198 Potekhina 1987).¹ 199

¹Unfortunately, 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 δ^{13} C and δ^{15} 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 ¹⁴C determinations in Table 2 are clearly due to the different dietary sources exploited by humans, combined with the occurrence of older 200 carbon in the fish samples (carp or pearl roach) (Lillie et al. 2011). In addition, as noted 201 elsewhere (e.g. Lake Baikal; Bronk Ramsey et al. 2014), the significant variability in δ^{13} C and 202 δ^{15} N values evidenced by the fish remains from the Dnieper (both archaeological and modern 203 samples) (Figure 4) indicates that δ^{13} C ratios are difficult to correlate to ¹⁴C offsets (Budd 2007; 204 Bronk Ramsey et al. 2014: 791). The δ^{13} C isotope ratios for the human samples at Dereivka 1 205 (male 40-50, cortical bone of skull) and Yasinovatka (male 30-40 cortical bone of skull) 206 are -22.4% and -22.6%, respectively. In isolation, these values would potentially suggest the 207 consumption of C_3 terrestrial proteins, given the values obtained from terrestrial fauna in this 208 region (Lillie et al. 2009, 2011), but in combination with the elevated $\delta^{15}N$ ratios exhibited by 209 these individuals and the archaeological δ^{15} N values of fish from the Dnieper (Figure 4), 210 the values clearly demonstrate the contribution of freshwater proteins into the diet (see also 211 Wood et al. 2013). 212

The δ^{15} N ratios of 12.7% at Dereivka 1 (male 40–50, cortical bone of skull) and 14.0% at 213 Yasinovatka (male 30-40 cortical bone of skull) (Table 2) would potentially indicate that 214 individual 54 at Yasinovatka is consuming proportionally higher quantities of freshwater 215 protein than terrestrial protein when compared to individual 29 at Dereivka 1. However, it is 216 recognized that considerable variability occurs within freshwater systems (Philippsen 2013; 217 Fernandes et al. 2014), and these two cemeteries are some distance apart within the Dnieper 218 system. Despite this, it is apparent that these differences in diet correlate with a larger offset 219 from the terrestrial baseline at Yasinovatka (472 yr) than the offset in evidence at Dereivka I 220 (251 yr). While linear interpolation is not viable on the basis of this data, in this example a 1.3%221 elevation in δ^{15} N at Yasinovatka (as compared to the Dereivka 1 sample) coincides with a 222 greater (i.e. 470 yr) offset from the terrestrial baseline sample when contrasted with the 12.7% 223 δ^{15} N ratio at Dereivka I (where a 250-yr offset is evident). It is anticipated that this variability 224 will be investigated in greater detail in future studies as at face value the higher human $\delta^{15}N$ 225 value at Yasinovatka equates to a larger FRE offset from the terrestrial baseline, but this is 226 complicated by the differences in fish $\delta^{13}C$ and $\delta^{15}N$ values between these two locations (Table 2) (Lillie et al. 2011).

Fauna and fish stable isotope values from burials within Yasinovatka and Dereivka indicate 228 that the terrestrial fauna have isotope values of -20.0% (δ^{13} C) and 7.5% (δ^{15} N) for a deer tooth 229 (incisoform, I2) in burial 28 at Yasinovatka and -20.4% (δ^{13} C) and 4.9% (δ^{15} N) for a deer 230 antler sample from Dereivka (Lillie et al. 2011). The variability between these samples is likely a 231 function of the two different elements analyzed, e.g. tooth dentine vs. antler collagen. Tooth 232 dentine forms incrementally over a period of months (Mitchell and Youngson 1969; Brown and 233 Chapman 1991; Sponheimer et al. 2003); therefore, the bulk dentine sample represents a mixed 234 dietary signal, including periods of nursing (see Balasse et al. 2001 for comparative research on 235 cattle dentine samples). Conversely, red deer antlers form at approximately 1 year of age 236 (Clutton-Brock 1982), and so reflect a postweaning phase of the diet. Therefore, one would 237 expect mature antler collagen to exhibit lower δ^{15} N values than a bulk dentine sample. 238

The archaeological fish tooth from burial 56 at Yasinovatka (Figure 4 - center) has values 239 of -21.1% (δ^{13} C) and 10.8% (δ^{15} N), while at Dereivka the fish from burials 5 and 46 240 have values of -16.8% and -25.5% for δ^{13} C and 14.3% and 10.5% for δ^{15} N, respectively 241 (Figure 4 – right and left sides of distribution) (Lillie et al. 2011:64–5). Along with the Neolithic 242 sample from Vil'nyanka (Figure 4, far left), these values clearly indicate a significant potential 243 for a wide range of offsets within a given cemetery population in the Dnieper Rapids region, 244 depending on which sources of dietary foodstuffs are being exploited (see for e.g. Rouja et al. 245 2003). In addition, there is clearly an established potential for significant intercemetery 246 variability both between individuals and chronological periods. 247

To date, stable isotope values have been recorded for the Epipaleolithic cemetery of Vasilyevka 248 III, where Lillie et al. (2003) analyzed 21 individuals (both males and females), all of whom had 249 δ^{15} N values over 11.9% (average = 12.7 ± 0.6%), with a maximum value of 14.1% recorded 250 on an adult male aged >55 yr, and δ^{13} C values between -22.5% and -21.6% (average = 251 $-22.2 \pm 1.5\%$). Faunal samples obtained during recent analyses have shown that at Mesolithic 252 sites in the region (e.g. Vyazivok, Igren VIII, and Rogalik 2) and further south in Crimea, $\delta^{15}N$ 253 values of 5.5% (Cervus elaphus) through to 8.8% (Sus scrofa) are evidenced (Lillie et al. 2011). 254 As such, the human isotope data are interpreted as indicating a relatively uniform diet, with 255 a strong dependence on freshwater and terrestrial animal protein. 256

Towards the latter part of the Mesolithic period, the cemetery sites of Vasilyevka II and 257 Marievka have produced contrasting indicators of diet, with Marievka (7000-6200 cal BC) 258 having an individual (burial 10, male, 35-45 yr) dated to 7020-6100 cal BC at 2σ (OxA-6200, 259 7620 ± 160 BP) with a δ^{13} C ratio of -21.7% and a δ^{15} N ratio of 13.0%, while two other indivi-260 duals (burials 4 [male? 50-60 yr]) dated to 7045-6690 cal BC (OxA-6199, 7955±55 BP) and 261 14 [indet. adult aged 35-45]), dated to 6685-6240 cal BC (OxA-6269, 7630±110 BP), 262 dated to 6685–6240 cal BC (OxA-6269, 7630 ± 110 BP) had δ^{13} C and δ^{15} N values of -22.0% 263 and -22.1% and 10.1% and 10.8%, respectively (all dates and isotope vales were obtained from 264 cortical bone from the skull of each individual) (Lillie et al. 2011). Given the available fish and 265 fauna values, it is clear that individuals 4 and 14 (adult male/adult of indeterminate sex) were 266 consuming diets that consisted principally of terrestrial resources, while individual 10 (adult male) 267 was consuming a diet with a stronger dependence on freshwater resources (Lillie et al. 2011:61-4). 268

At the later Mesolithic site of Vasilyevka II, dated to ~7300–6220 cal BC, analysis of 14 individuals by Lillie and Jacobs (2006) led to the observation that both males and females 270

consumed diets that were rich in freshwater resources, with δ^{13} C values ranging from -21.8 to -20.1% (-20.9 ± 0.5%) and δ^{15} N ratios ranging from 12.4 to 14.7% (13.4 ± 0.7%). 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). 276

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For the Neolithic period, the cemeteries of Vovnigi I, Yasinovatka, Dereivka 1, 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 δ^{13} C value of -20.4% and a δ^{15} N ratio of 4.9%, while a deer tooth sample from Yasinovatka has produced ratios of -20.0% and 7.4% for δ^{13} C and δ^{15} N, respectively, and a deer tooth sample from Vovnigi I produced ratios of -19.1% and 6.9% for δ^{13} C and δ^{15} N, respectively. The Neolithic fauna from Molukhov Bugor produced ranges between -23.2% (*Cervus elaphus*) to -18.9% (*Ursus arctos*) for δ^{13} C and 4.8% (*Sus scrofa*) to 7% (*Ursus arctos*) for δ^{15} 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% and -19.6% for 287 δ^{13} C (-22.7 ± 1.2%) and 12.2% to 14.3% for δ^{15} N (13.4 ± 0.7%). These ratios are 288 clearly elevated when compared to the contemporary fauna, and the situation is the same at 289 Yasinovatka where values of -24.1% to -22.4% (average = $-23.4\pm0.6\%$) and 11.4% to 290 15.1% (average = $13.6 \pm 1.1\%$) are recorded for δ^{13} C and δ^{15} N, respectively. At Dereivka I, 291 values of -23.6% to -21.7% (average $= -23.1 \pm 0.7\%$) and 10.0% to 13.5% ($11.6 \pm 1.3\%$) 292 are recorded for δ^{13} C and δ^{15} N, respectively. The values for the humans are difficult to interpret 293 due to the variable faunal ranges for the period, although it is apparent that freshwater 294 resources (fish) are contributing some proportion of the dietary proteins at these sites. This is 295 reinforced by the fish values from Dereivka I, Vil'nyanka, and Yasinovatka, which 296 exhibit very wide ranges for δ^{13} C, between -26% and -16.8% and δ^{15} N ratios of 10% to 297 14.3%. While limited in number, the available human bone samples from Vasilyevka V have 298 produced dietary isotope compositions with δ^{13} C ranging between -23.2% and -20.2% and 299 δ^{15} N between 10.0% and 12.3%, again the isotope values of certain individuals are indicative 300 of the consumption of freshwater resources. Interestingly, while the two $\delta^{15}N$ ratios of 10.0% 301 and 10.6% at Vasilyevka V might suggest that these individuals had a mainly terrestrial 302 resource oriented diet, the δ^{13} C values of -22.4% and -23.2% would lend weight to the 303 observation that freshwater resources were being consumed. The single individual with an 304 elevated δ^{15} N ratio of 12.3% probably consumed a diet with a higher input of freshwater 305 resources, and the δ^{13} C ratio of -22.3% for this individual reinforces this observation 306 (Lillie et al. 2011:65). 307

During the Eneolithic period at the site of Molukhov Bugor, which is located higher up 308 in the Dnieper River catchment, the stable isotope data are broadly consistent with the 309 Neolithic data, although only four human samples were available for analysis from this 310 location. The human samples range between -23.1% and -21.2% (average $= -21.8 \pm 1\%$) for 311 δ^{13} C and 11.5% to 12.9% (average = 12.2 ± 0.7%) for δ^{15} N. The Eneolithic faunal samples 312 from Molukhov Bugor exhibit a range of -22.6% to -19.8% (average = $-21.5 \pm 1.1\%$) 313 for δ^{13} C and 5.6% to 7.5% (average = $6.7 \pm 0.7\%$) for δ^{15} N. As such, it is also likely that 314 freshwater resources, alongside terrestrial herbivores, contributed to the Eneolithic diet at 315 Molukhov Bugor. 316

THE PROBLEM WITH FISH

In summary, it is clear from the isotope data and archaeological record that the inclusion of fish 317 protein in prehistoric diets was a major factor in the Dnieper Rapids region. Returning to the 318 core study themes of this paper, it is evident from the δ^{13} C and δ^{15} N values that (i) in most cases 319 freshwater protein contributed a large (if not major) proportion of the diet for the prehistoric 320 populations throughout the Epipaleolithic to Eneolithic periods. In addition, (ii) the amount of 321 freshwater protein appears to be largely consistent throughout the chronological phases; the 322 earliest dated site in the sequence, i.e. Vasilyevka III (at ~10,000 cal BC) has clear evidence for 323 the consumption of freshwater protein (Lillie et al. 2003), and this continues through to the 324 Eneolithic/Copper Age at Igren VIII, up to ~3000 cal BC, although the pattern for freshwater 325 protein consumption exhibits variation at both the inter- and intrasite levels of analysis. Finally, 326 the ¹⁴C determinations of human, fauna, and fish collagen samples recovered in close associa-327 tion, demonstrate very convincing evidence for a freshwater reservoir effect, ranging between 328 \sim 250 and 470 vr, dependent on the feeding habits of the species of fish consumed (benthic versus 329 bottom feeders, etc.) and the proportion of fish protein consumed in an individual's diet. 330

A fundamental problem with this approach to paleodietary reconstruction lies in the fact that 331 the fish isotope ratios are not consistent at the intra- and interspecies levels (fish can be deep or 332 shallow water feeders, predators, and benthic feeders), and the ethnographic evidence for 333 selectivity in human consumption patterns of fish would seem to negate any easy linear 334 interpolation of the nitrogen ratios that are in evidence. The stable isotope data for 335 individual 8–74/4 from Igren VIII (8744 in Figure 2) would suggest that, in light of the available 336 fish ratios from the Dnieper Rapids (Figure 4), this individual could either have placed a very 337 particular dietary focus on a single species, or was consuming a diet that differed markedly to 338 the later (Eneolithic) individuals at this location. This individual appears anomalous in 339 relation to the individuals from the Dnieper Rapids region during the prehistoric period, 340 and recent dating of this individual has indicated that as opposed to being of Neolithic date, 341 individual 4 from Igren VIII is in fact an intrusive interment.² While the consumption of 342 freshwater resources occurs throughout the prehistoric period, we can postulate that the low 343 δ^{15} N values at Igren VIII (e.g. individual 8–78/24 [87824 in Figure 2]) might indicate that there 344 are some human individuals with less potential for a reservoir offset. This could be assessed 345 using associated faunal remains from burials, thus potentially offering a way to limit the biases 346 that occur when dating these populations. 347

In conclusion, it is clear from the dating and isotope studies undertaken to date (e.g. Lillie et al. 348 2009, 2011) that the majority of dates on individuals with elevated nitrogen ratios (i.e. above 349 $\sim 12\%$) should be considered to have a potential reservoir effect. As noted above, at $\sim 12.7\%$ at 350 Dereivka this is a 250-yr offset from the terrestrial baseline (the issue of limited faunal values 351 not withstanding), while at 14.0% at Yasinovatka the offset rises to 470 yr. At this stage in the 352 research agenda, it is clear that targeted excavations aimed at recovering a combination of 353 human, fauna, and where possible, fish samples, are needed in order to adequately determine 354 potential degrees of offset, and the actual offset in ¹⁴C yr, alongside the stable isotope values for 355 the populations studied (e.g. Schulting et al. 2014). 356

²Since the initial study was undertaken, AMS dating of individual 8–74/4 from Igren VIII has shown that this individual in 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 VIIII (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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REFERENCES

- Balasse M, Bocherens H, Mariotti A, Ambrose SH. 2001. Detection of dietary changes by intratooth carbon and nitrogen isotopic analysis: an experimental study of dentine collagen of cattle (*Bos taurus*). Journal of Archaeological Science 28(3):235–45.
- Beaumont J, Gledhill A, Lee-Thorp J, Montgomery J. 2012. Childhood diet: a closer examination of the evidence from dental tissues using stable isotope analysis of incremental human dentine. *Archaeometry* 55(2):277–95.
- Brinch Petersen E, Meiklejohn C. 1995. Paradigm lost: searching for "complexity" in the Mesolithic. Paper read at the conference From the Jomon to Star Carr: International Conference on Holocene Hunter–Gatherers in Temperate Eurasia. Cambridge/Durham, England, September 1995.
- Bronk Ramsey C, Schulting R, Goriunova OI, Bazaliiskii VI, Weber AW. 2014. Analyzing radiocarbon reservoir offsets through stable nitrogen isotopes and Bayesian modeling: a case study using paired human and faunal remains from the Cis-Baikal region, Siberia. *Radiocarbon* 56(2):789–99.
- Brown WAB, Chapman N. 1991. The dentition of red deer (*Cervus elaphus*): a scoring scheme to assess age from wear of the permanent molariform teeth. *Journal of Zoology* 224(4):519–36.
- Budd CE. 2007. The Dnieper Rapids region of the Ukraine: a re-assessment of prehistoric chronology and subsistence strategies [unpublished Master's thesis]. Oxford University.
- Clutton-Brock TH. 1982. *Red Deer: Behaviour and Ecology of Two Sexes*. Chicago: University of Chicago Press.
- Demirjian A, Levesque GY. 1980. Sexual differences in dental development and prediction of emergence. Journal of Dental Research 59(7):1110–22.
- DeNiro M. 1985. Postmortem preservation and alteration of *in vivo* bone collagen isotope ratios in relation to palaeodietary reconstruction. *Nature* 317(6040):806–9.
- Fernandes R, Rinne C, Nadeau M-J, Grootes P. 2014. Towards the use of radiocarbon as a dietary proxy: establishing a first wide-ranging radiocarbon reservoir effects baseline for Germany. *Environmental Archaeology: The Journal of Human Palaeoecology.* DOI: 10.1179/1749631414Y.0000000034.
- Goodman AH, Martin DL, Armelagos GJ, Clark G. 1984. Indications of stress from bones and teeth. In: Cohen MN, Armelagos GJ, editors. *Palaeopathology at the Origins of Agriculture*. Orlando: Academic Press. p 13–49.

- Gorobets LV, Matlaev IV. 2014. Birds from the Old East Slavic settlement "Igren 8" (12th–13th century AD; Ukraine). Visn. Dnipropetr. Univ. Ser. Biol. Ekol. [Bulletin of Dnipropetrovsk University. Biology, Ecology] 22(1):66–70.
- Hedges REM, Reynard LM. 2007. Nitrogen isotopes and the trophic level of humans in archaeology. *Journal of Archaeological Science* 34(8):1240–51.
- Henderson RC. 2015. Early life histories: a study of past childhood diet and health using stable isotopes and enamel hypoplasia [unpublished PhD thesis]. Oxford: Oxford University.
- Henderson RC, Lee-Thorp J, Loe L. 2014. Early life histories of the London poor using δ^{13} C and δ^{15} N stable isotope incremental dentine sampling. *American Journal of Physical Anthropology* 154(4):585–93.
- Lillie MC. 2008. Suffer the children: 'visualising' children in the archaeological record. In: Bacvarov K, editor. Babies Reborn: Infant/Child Burials in Pre- and Protohistory. BAR Reports (International Series) 1832. Oxford: Archaeopress. p 33–43.
- Lillie MC, Jacobs K. 2006. Stable isotope analysis of fourteen individuals from the Mesolithic cemetery of Vasilyevka II, Dnieper Rapids region, Ukraine. *Journal of Archaeological Science* 33(6):880–6.
- Lillie MC, Richards MP. 2000. Stable isotope analysis and dental evidence of diet at the Mesolithic– Neolithic transition in Ukraine. *Journal of Archaeological Science* 27(10):965–72.
- Lillie MC, Richards MP, Jacobs K. 2003. Stable isotope analysis of 21 individuals from the Epipalaeolithic cemetery of Vasilyevka III, Dnieper Rapids region, Ukraine. *Journal of Archaeological Science* 30(6):743–52.
- Lillie MC, Budd CE, Potekhina ID, Hedges REM. 2009. The radiocarbon reservoir effect: new evidence from the cemeteries of the Middle and Lower Dnieper Basin, Ukraine. *Journal of Archaeological Science* 36(2):256–64.
- Lillie MC, Budd CE, Potekhina ID. 2011. Stable isotope analysis of prehistoric populations from the cemeteries of the Middle and Lower Dnieper Basin, Ukraine. *Journal of Archaeological Science* 38(1):57–68.
- Longin R. 1971. New method of collagen extraction for radiocarbon dating. *Nature* 230(5291): 241–2.
- Massler M, Schour I, Poncher HG. 1941. Developmental pattern of the child as reflected in the calcification pattern of the teeth. *American Journal of Diseases of Children* 62(1):33–67.

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- Meiklejohn C, Brinch Petersen E, Babb J. 2009. From single graves to cemeteries: an initial look at chronology in Mesolithic burial practice. In: McCartan S, Schulting R, Warren G, Woodman P, editors. *Mesolithic Horizons*. Oxford: Oxbow Books. p 639–49.
- Mitchell B, Youngson RW. 1969. Teeth and age in Scottish red deer–a practical guide to the determination of age. Appendix. In: *The Red Deer Commission Annual Report for 1968*. London: Her Majesty's Stationery Office. p 1–3.
- Phiilippsen B. 2013. The freshwater reservoir effect in radiocarbon dating. *Heritage Science* 1(24): 1–19.
- Rouja PM, Dewailly É, Blanchet C, the Bardi Community. 2003. Fat, fishing patterns, and health among the Bardi people of north western Australia. *Lipids* 38(4):399–405.
- Schulting RJ, Bronk Ramsey C, Bazaliiskii VI, Goriunova OI, Weber A. 2014. Freshwater reservoir offsets investigated through paired humanfaunal ¹⁴C dating and stable carbon and nitrogen isotope analysis at Lake Baikal, Siberia. *Radiocarbon* 56(3):991–1008.
- Schwarcz HP, Schoeninger MJ. 1991. Stable isotope analysis in human nutritional ecology. *Yearbook* of Physical Anthropology 34(S13):283–321.
- Sellen DW, Smay DB. 2001. Relationship between subsistence and age at weaning in "preindustrial" societies. *Human Nature* 12(1):47–87.
- Sponheimer M, Robinson T, Ayliffe L, Roeder B, Hammer J, Passey B, West A, Cerling T, Dearing D, Ehleringer J. 2003. Nitrogen isotopes in mammalian

herbivores: hair $\delta^{15}N$ values from a controlled feeding study. *International Journal of Osteoarchaeology* 13(1–2):80–7.

- Telegin DYa. 1986. Dereivka: A Settlement and Cemetery Of Copper Age Horse Keepers on the Middle Dnieper. Oxford: BAR International Series. Volume 287. Oxford: Archaeopress.
- Telegin DYa, Filenko OS. 1982. Могилник среднестоговской културы в днеровском надпорожьу (A Sredniy Stog burial ground in the upper Dnieper Rapids region). Sovetskaya Archeologia 1:80–7.
- Telegin DYa, Potekhina ID. 1987. Neolithic Cemeteries and Populations in the Dnieper Basin. British Archaeological Reports International Series. Volume 383. Oxford: Archaeopress.
- Telegin DYa, Zaliznyak LL. 1975. 'Raskopki na Igrenskom poluostrove' [Excavations at the Igren Peninsula]. *Arkheologicheskie Otkrytiya* 1974:358–9.
- Telegin DYa, Potekhina ID, Lillie MC, Kovaliukh MM. 2002. The chronology of the Mariupol-type cemeteries of Ukraine re-visited. *Antiquity* 76(292):356–63.
- Telegin DYa, Lillie MC, Potekhina ID, Kovaliukh MM. 2003. Settlement and economy in Neolithic Ukraine: a new chronology. *Antiquity* 77(297):456–70.
- Wood RE, Higham TFG, Buzilhova A, Suvorov A, Heinemeier J, Olsen J. 2013. Freshwater radiocarbon reservoir effects at the burial ground of Minino, northwest Russia. *Radiocarbon* 55(1):163–77.

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