Radiocarbon, Vol 58, Nr 2, 2016, p 267–289 DOI:10.1017/RDC.2016.13

STONE AGE POTTERY CHRONOLOGY IN THE NORTHEAST EUROPEAN FOREST ZONE: NEW AMS AND EA-IRMS RESULTS ON FOODCRUSTS

Henny Piezonka[1*] • John Meadows[2] • Sönke Hartz[3] • Elena Kostyleva[4] •
Nadezhda Nedomolkina[5] • Marina Ivanishcheva[6] • Natalya Kosorukova[7] • Thomas Terberger[8]

[1]German Archaeological Institute, Eurasia Department, Berlin, Germany.
[2]Center for Baltic and Scandinavian Archaeology, Foundation of the Schleswig-Holstein State Museums, Schloss Gottorf, Schleswig, Germany; also Leibniz Laboratory for Radiometric Dating and Stable Isotope Research, Christian Albrechts University Kiel, Germany.
[3]Archaeological State Museum, Foundation of the Schleswig Holstein State Museums, State Museums Schloss Gottorf, 24837 Schleswig, Germany.
[4]Ivanovo State University, Ivanovo, Russia.
[5]Vologda State Museum for History, Architecture and Art, Vologda, Russia.
[6]Child and Youth Centre "Lider," Vologda, Russia.
[7]Cherepovec State University and Cherepovec Museum Association, Cherepovec, Russia.
[8]Lower Saxony State Agency for Heritage Services, Hanover, Germany.

ABSTRACT. Pottery produced by mobile hunter-gatherer-fisher groups in the northeast European forest zone is among the earliest in Europe. Absolute chronologies, however, are still subject to debate due to a general lack of reliable contextual information. Direct radiocarbon dating of carbonized surface residues ("foodcrusts") on pots can help to address this problem, as it dates the use of the pottery. If a pot was used to cook fish or other aquatic species, however, carbon in the crust may have been depleted in ^{14}C compared to carbon in terrestrial foods and thus appear older than it really is (i.e. showing a "freshwater reservoir effect," or FRE). A connected problem, therefore, is the importance of aquatic resources in the subsistence economy, and whether pots were used to process aquatic food. To build better chronologies from foodcrust dates, we need to determine which ^{14}C results are more or less likely to be subject to FRE, i.e. to distinguish crusts derived mainly from aquatic ingredients from those composed mainly of terrestrial foods. Integrating laboratory analyses with relative chronologies based on typology and stratigraphy can help to assess the extent of FRE in foodcrust dates. This article reports new ^{14}C and stable isotope measurements on foodcrusts from six Stone Age sites in central and northern European Russia, and one in southeastern Estonia. Most of these ^{14}C results are not obviously influenced by FRE, but the isotopic data suggest an increasing use of aquatic products over the course of the 6th and 5th millennia cal BC.

KEYWORDS: foodcrusts, pottery, carbon and nitrogen stable isotopes, freshwater reservoir effects, hunter-gatherer-fisher societies, northeast Europe.

INTRODUCTION

Pottery produced by mobile hunter-gatherer-fisher groups in the northeast European forest zone is among the earliest in Europe, probably appearing by the end of the 7th millennium cal BC in some regions (Hartz et al. 2012; Mazurkevich and Dolbunova 2012; Vybornov et al. 2012). The emergence, dispersal, and further development of these early ceramic traditions are of great relevance also for central European questions. The earliest ceramic vessels in the east emerged in a cultural environment that was based on a foraging economy and seasonal mobility, while in southern, central, and western Europe the earliest pottery is mostly associated with the transition towards a productive economy, residential sedentism, and the emergence of more complex forms of society. In the eastern research tradition, pottery is seen as the main defining marker of the Neolithic period (Oshibkina 2006), while in western archaeology a different definition of the Neolithic based on a food-producing economy is preferred (Scharl 2004). This article follows the local, eastern terminology. The fact that hunter-gatherer ceramic traditions have not only left their traces in eastern Europe but probably also reached west as far as northern Germany and southern Scandinavia, where they influenced the adoption of pottery in the Ertebølle culture, has increasingly been understood and triggered an immense interest in this complex (see e.g. Jordan and Zvelebil 2009; Hartz et al. 2011; Povlsen 2013).

---

*Corresponding author. Email: Henny.Piezonka@dainst.de.

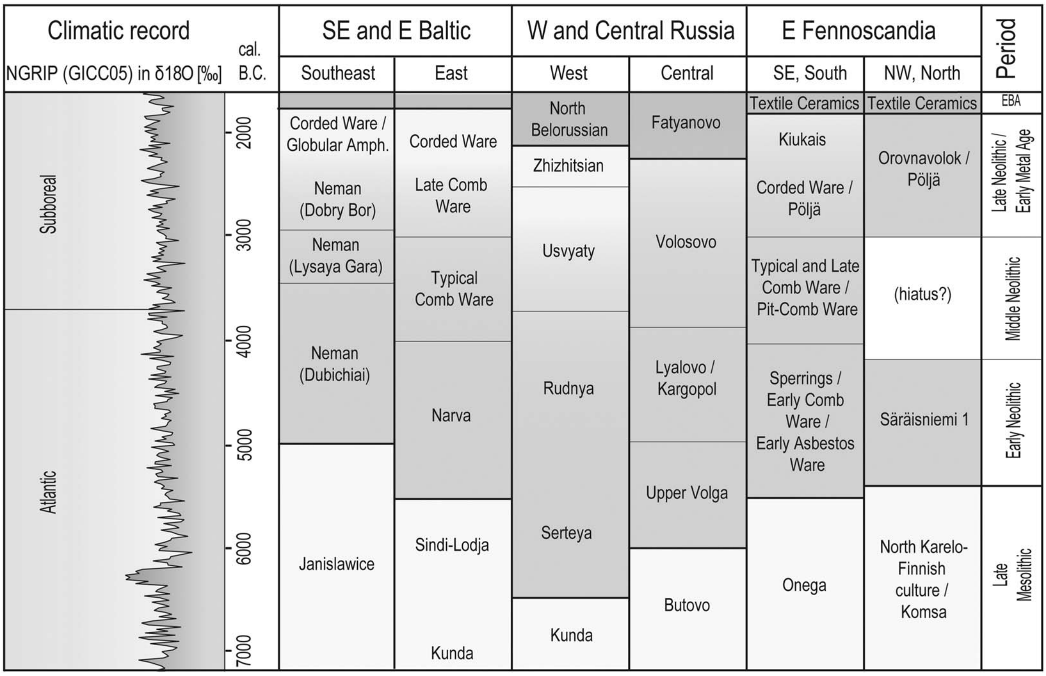

Figure 1 Sequence of archaeological cultures in northeastern Europe from the Late Mesolithic to the Early Metal Age. The onset of the Early Neolithic is defined, according to the eastern scientific tradition, by the first appearance of pottery vessels in the archaeological record, initial evidence for domesticates is indicated by shading (illustration: H Piezonka).

Stratigraphic observations and the typological evolution of pottery styles provide the main tools for building relative archaeological chronologies. On this basis, regional sequences of prehistoric cultural units have been worked out in more or less detail for various parts of northeastern Europe (for an overview see Piezonka 2015) (Figure 1). Absolute chronologies, however, are still subject to debate due to a general lack of dependable dates and reliable contextual information. Therefore, one of the foremost tasks in current research is to build a reliable chronological framework for the emergence and further evolution of early ceramics and their cultural contexts. A second set of questions concerns the use of early pottery. Connected to this are more general questions of economic developments, and in particular the unsolved problem of when food production (animal husbandry and arable farming) was introduced in the various parts of the northeastern European forest zone.

Until relatively recently, the absolute chronology of the appearance, dispersal, and evolution of early pottery in this region was based on radiometric radiocarbon measurements, often with large uncertainties, from samples such as charcoal, wood, and organic sediment, found in questionable temporal association with the pottery concerned. Procedures have been developed to date carbon found in the pottery fabric itself, by dissolving the matrix and combusting the residue, with frequently plausible but not always convincing results (Zaitseva et al. 2009). The results are often less precise than those from samples of wood and organic sediment, and more importantly, any organic carbon present would contribute to the ^{14}C age, not only food residues, soot, or organic temper (Karmanov et al. 2014:736).

Direct accelerator mass spectrometry (AMS) ^{14}C dating of carbonized foodcrusts on pottery appears to solve many of the potential problems (Piezonka 2008), but forces us to confront another: that carbon in freshwater food chains is often subject to large and variable reservoir effects, which may be expected to lead to freshwater reservoir effects (FRE) in some foodcrust dates. The issue cannot be ignored, as it is clear from archaeological evidence that fishing was an important part of the subsistence economy among the earliest pottery-using communities (see below), and FRE have been demonstrated in human remains in this region (e.g. Wood et al. 2013). If fish was cooked in pots, therefore, some ^{14}C-depleted carbon is likely to have been incorporated in foodcrusts.

In order to obtain accurate absolute chronologies for early pottery from the ^{14}C dating of carbonized food remains, we need to establish which ^{14}C results are potentially subject to reservoir effects and to what extent. Various tools can address this question: (1) discrepancies between calibrated ^{14}C ages and the relative chronology of the samples based on archaeological information (typology, stratigraphy) can suggest which samples, if any, have a higher risk of FRE; (2) paired samples of different materials can be dated (e.g. carbonized surface residue and terrestrial macroremains embedded in it); (3) the percentage of carbon in each sample derived from aquatic sources can be estimated from elemental analysis-isotope ratio mass spectrometry (EA-IRMS) results (%C, %N, δ^{13}C, and δ^{15}N), if these parameter values are sufficiently different in terrestrial and aquatic foods, and if charring and diagenesis do not mask these differences; (4) qualitative and semi-quantitative analytical techniques (microscopy and biomolecular analyses) may be used to attribute components of the foodcrust to terrestrial or aquatic sources.

In this paper, we focus on isotopic signals and elemental concentrations in ^{14}C-dated foodcrusts from the stratified complexes of the northern Russian Stone Age sites of Veksa 3, Karavaikha 1 and 4, and Tudozero 5, and Estonian material from Kääpa. In addition, new isotopic results from the central Russian sites of Sakhtysh 2a and Ozerki 17 will be discussed against the background of the already published AMS dates of the same samples (see Hartz et al. 2012).

MATERIALS AND METHODS

Stone Age Pottery from the Forest Zone of Northeastern Europe

Veksa 3, Russia

The pre- and early-historic settlement of Veksa 3 is a pivotal site with regard to the cultural development of northwestern European Russia. Located in the upper Sukhona basin, ~20 km east of the provincial capital of Vologda (Figure 2), the site extends along the left bank of River Vologda. The exceptional importance of Veksa 3 is due to the clearly stratified sequence of archaeological layers spanning 8 millennia (Nedomolkina 2004; Lorenz et al. 2012). Foodcrusts on eight Early and Middle Neolithic pottery vessels from the Veksa 3 section of the site have been sampled for the present paper (Table 1; Figures 2 and 3). Although most of these sherds are from surface collections, they can be associated with the respective stratigraphic units on the basis of their typology (Piezonka 2015: Figure 41). Of the vessels investigated, two were ^{14}C dated previously, and %C, %N, δ^{13}C, and δ^{15}N have now been measured in the dating extracts (KIA-33927; KIA-33928; Piezonka 2008). Of one vessel of which the interior charred crust had been dated previously (KIA-33926; Piezonka 2008), we have now dated the outer crust (KIA-49796), and obtained EA-IRMS results from both dating extracts. Three new sherds (KIA-49797, KIA-49798, KIA-49799; Piezonka 2015) are dated here for the first time, and EA-IRMS results are reported for two further foodcrusts, which were unfortunately too small to date (KIA-49789, KIA-49790; Piezonka 2015). Charcoal from a pit in layer 9, the lowest cultural horizon, was also dated, to provide a *terminus post quem* for the entire sequence (KIA-33929; Piezonka 2008).

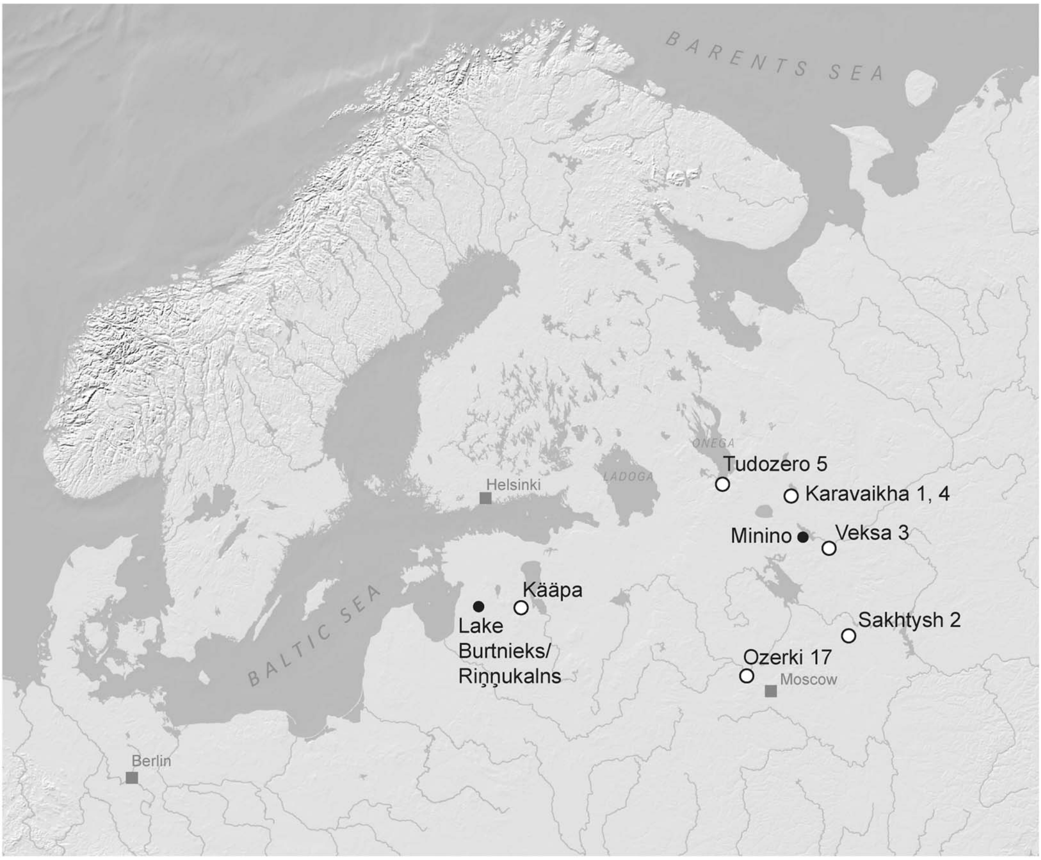

Figure 2 Sites in northeastern Europe with prehistoric ceramics from which organic residue on pottery was analyzed (white dots) and other sites mentioned in the text (black dots) (illustration: H Piezonka).

Karavaikha 1 and 4, Russia

The archaeological complex of Karavaikha is situated in the north of Vologda province on the banks of the River Eloma, a few kilometers upstream from its mouth at Lake Vozhe (Figure 2). At Karavaikha 4, excavations have revealed a lower cultural horizon with well-preserved wooden constructions probably connected to fishing activities; associated finds include a small number of pot sherds (Kosorukova 2007; Kiryanova and Kosorukova 2013). Although this lower cultural horizon has been regarded as a closed context, stemming from a confined episode of human activity in the Early Neolithic, conventional ^{14}C dates on samples attributed to this layer span a long period from the beginning of the 6th to the first third of the 5th millennium cal BC. Foodcrusts of two vessels were investigated for the present study, among them one vessel resembling comb-decorated ware of the middle Upper Volga culture (AAR-17172) and one typologically less specific vessel, decorated with various imprints of natural materials (AAR-17171). In addition, one animal bone from this complex was dated (AAR-17170) (Table 1, Figure 4). A second cultural horizon is thought to belong to a later phase of the Stone Age; two conventional ^{14}C dates cover the end of the 6th and the beginning of the 5th millennium cal BC. At Karavaikha 1, on the opposite bank of the river, a Neolithic–Early Metal Age cemetery and settlement remains from various periods have been excavated (Utkin and Kostyleva 2001). Recent test trenches at this site have yielded settlement evidence of the

Table 1 EA-IRMS and AMS results on organic material from Stone Age sites in northwest European Russia and Estonia. Previously unpublished data, except for † (^{14}C age from Piezonka 2008) and ‡ (^{14}C age from Hartz et al. 2012).

Site	Sample no. #	Context	Typological association	Material	Lab nr	Yield (%)*	%C§	%N§	atomic C/N	δ^{13}C (‰)§	δ^{15}N (‰)§	Conventional ^{14}C age BP*	Maximum age cal BC^ (95.4% probability)
Veksa 3	Ve-2007/soil 1	excavation 2002, layer 9, pit	Early Neolithic	organically enriched soil	KIA-33929	18	62*			–24.3*		6340 ± 30†	5460–5220
Veksa 3	Ve-2007/114	1996, surface find on river bank	Early Neolithic (Upper Volga)	foodcrust	KIA-49797	68	60.3	4.0	17.5	–27.38	8.05	6386 ± 21	5470–5310
Veksa 3	Ve-2007/115	surface find on river bank	Early Neolithic (Earliest Comb-Pitted ware)	foodcrust	KIA-49798	62	51.4	5.0	12.1	–27.76	6.31	6314 ± 22	5340–5220
Veksa 3	Ve-2007/112	1996, surface find on river bank	Early Neolithic ("2nd comb ceramic complex")	foodcrust	KIA-49799	62	54.1	54.1	7.7	–28.10	9.42	6285 ± 30	5320–5210
Veksa 3	Ve-2007/111	1996, surface find on river bank	Early Neolithic ("2nd comb ceramic complex")	foodcrust	KIA-33927	71	53.4	8.7	7.2	–29.34	10.27	6185 ± 30†	5230–5040
Veksa 3	Ve-2007/117	1996, surface find on river bank	Late Early Neolithic ("Northern types")	foodcrust	KIA-33928	61	56.3	4.7	14.1	–30.88	10.33	6105 ± 30†	5210–4930
Veksa 3	Ve-2007/118b	1996, surface find on river bank (shore segment 6)	Middle Neolithic (Narva)	foodcrust (outer surface)	KIA-49796	52	56.3	9.4	7.0	–28.54	11.87	5492 ± 23	4440–4260
Veksa 3	Ve-2007/118a	1996, surface find on river bank (shore segment 6)	Middle Neolithic (Narva)	foodcrust (inner surface)	KIA-33926	61	48.8	9.2	6.2	–30.51	12.66	5425 ± 30†	4350–4230
Veksa 3	Ve-2007/104	excavation 2001, squ. 167-K, layer 6	Middle Neolithic (Comb-Pitted ware)	foodcrust	KIA-49789	17	46.6	8.3	6.6	–30.59	13.35	no date	no date
Veksa 3	Ve-2007/106	excavation 2001, squ. 167-K, layer 6	Middle Neolithic (Comb-Pitted ware)	foodcrust	KIA-49790	16	36.5	5.0	8.5	–31.68	11.61	no date	no date
Karavaikha 4	Ka4-2012/bone1	2008, horizon 8, sq. F10, x0.95m, y 0.40 m		indeterminate ungulate bone dagger	AAR-17170				3.2	–21.72	4.6	7009±40	5990–5790
Karavaikha 4	Ka4-2012/5	2006, horizon 8, trench 7 sq. Y-25	Early Neolithic	foodcrust	AAR-17172		36.6	6.7	6.5	–25.74	10.21	6672±31	5650–5530
Karavaikha 4	Ka4-2012/1	2007, horizon 5, sq. O-19, find no. 14027/34	Early Neolithic	foodcrust	AAR-17171		57.2	3.2	20.9	–26.15	0.89	6222±30	5300–5060

Table 1 (*Continued*)

Site	Sample no. #	Context	Typological association	Material	Lab nr	Yield (%)*	%C§	%N§	atomic C/N	δ^{13}C (‰)§	δ^{15}N (‰)§	Conventional ^{14}C age BP*	Maximum age cal BC^ (95.4% probability)
Karavaikha 1	Ka1-2012/1	2002, test trench 1, find no. 3942	Middle Neolithic (Kargopol')	foodcrust	AAR-17169		16.4	3.5	5.5	–27.90	9.75	5588±32	4490–4350
Tudozero 5	Tu-2012/3	1990 EN horizon lower black layer house	Early Neolithic (earliest Comb ware)	foodcrust	AAR-17174		17.3	2.0	10.1	–27.24	10.68	6660±32	5640–5530
Tudozero 5	Tu-2012/2	1989 EN horizon house pit	Early Neolithic (Sperrings)	foodcrust	AAR-17173		48.9	6.6	8.6	–26.95	13.89	6241±30	5310–5070
Sakhtysh 2a		Sq. 25, depth 2.49 m, layer IIg (same sherd as KIA-39301)	Early Neolithic (early Upper Volga)	plant (willow bast string embedded in foodcrust)	KIA-39300	37	—	—	—	–26.88	—	6847±31‡	5800–5660
Sakhtysh 2a		Sq. 25, depth 2.49 m, layer IIg (same sherd as KIA-39300)	Early Neolithic (early Upper Volga)	foodcrust	KIA-39301	53	58.2	3.0	19.3	–25.76	3.40	6860±31‡	5840–5660
Sakhtysh 2a		Trench 2004, sq. 18, depth 2.48 m, layer IIg	Early Neolithic (early Upper Volga)	foodcrust	KIA-39308	50	58.0	9.6	6.1	–21.53	6.72	7018±45‡	6000–5790
Sakhtysh 2a		Trench 2004, sq. 11, depth 2.44 m, layer IIg	Early Neolithic (early Upper Volga)	foodcrust	KIA-39309	46	36.0	5.8	6.1	–20.80	5.36	7037±27‡	6000–5840
Sakhtysh 2a		Trench 2004, sq. 25, depth 2.94 m, layer IIg	Early Neolithic (early Upper Volga)	foodcrust	KIA-39310	46	66.5	9.3	7.2	–29.79	10.73	7356±30‡	6360–6090
Sakhtysh 2a		Trench 1999, sq. 14, depth 2.66 m, layer IIg	Early Neolithic (early Upper Volga)	foodcrust	KIA-39311	36	67.9	10.2	6.6	–23.11	5.88	7072±36‡	6020–5880
Sakhtysh 2a		Trench 2004, sq. 32, depth 2.23 m, layer IIb	Early Neolithic (developed Upper Volga)	foodcrust	KIA-39303	46	58.0	8.7	6.6	–23.50	7.80	6348±26‡	5470–5220
Sakhtysh 2a		Trench 2004, sq. 29, depth 2.58 m, layer IIg	Early Neolithic (developed Upper Volga culture)	foodcrust	KIA-39312	51	48.9	2.6	18.97	–24.50	5.80	6395±28‡	5470–5310
Sakhtysh 2a				foodcrust	KIA-39313	29	45.2	1.8	25.7	–24.57	5.96	6371±30‡	5470–5300

		Trench 2004, layer IIg	Early Neolithic (developed Upper Volga)										
Sakhtysh 2a		Trench 2004, sq. 32, depth 2.13 m, layer IIb	Early Neolithic (late Upper Volga)	foodcrust	KIA-39302	53	66.1	2.5	26.2	–25.34	3.23	6160±27‡	5220–5030
Sakhtysh 2a		excavation 1993, grave no. 66, find no. 704	Early Metal Age (Volosovo culture)	elk (*Alces alces*) tooth pendant	AAR-21042		31.1	11.7	3.2	–21.2	4.9	5252±29	4230–3970
Ozerki 17		Excavations 1991-1993, layer III	Early Neolithic (developed Upper Volga)	foodcrust	KIA-39306	40	66.2	6.5	10.1	–27.70	8.25	6369±27‡	5470–5300
Ozerki 17		Trench 1992, sq. 38, depth 2.30-2,32 m, layer II	Middle Neolithic (early Lyalovo)	foodcrust	KIA-39307	36	59.6	8.6	7.0	–25.50	8.39	5693±29‡	4610–4450
Kääpa	Kä-2007/56		Early Neolithic (Narva)	foodcrust	KIA-35897	44	52.9	8.5	7.2	–30.61	8.37	6540±40†	4990–4780
Kääpa	Kä-2007/36		Early Neolithic (Narva)	foodcrust	KIA-49794	63	66.4	7.8	9.9	–29.41	10.31	6320±30	5370–5220
Kääpa	Kä-2007/45		Early Neolithic (Narva)	foodcrust	KIA-49795	54	60.6	5.8	12.3	–29.09	8.03	6219±25	5300–5060
Kääpa	Kä-2007/62		Early Neolithic (Narva)	foodcrust	KIA-49793	74	57.7	7.8	8.7	–29.88	12.09	6015±35	4840–4720
Kääpa	Kä-2007/34		Early Neolithic (Narva)	foodcrust	KIA-33921	57	57.6	9.8	6.8	–30.12	11.76	5985±35†	5620–5380
Kääpa	Kä-2007/26		Early Neolithic (Narva)	foodcrust	KIA-49792	49	62.6	9.2	7.9	–33.16	10.94	5798±21	4720–4580
Kääpa	Kä-2007/70		Early Neolithic (Narva)	lamp charred organic residue	KIA-49791	49	57.6	8.3	8.1	–29.20	10.67	—	—

---

§EA-IRMS measurements from the Natural History Museum, Berlin, Germany (Ozerki 17, Sakhtysh 2a), School of Life Sciences, University of Bradford, England (Kääpa, Veksa 3) and AMS ^{14}C Dating Centre at Aarhus University, Denmark (Karavaikha, Tudozero).

*Measurements from the Leibniz Labor for AMS Dating and Stable Isotope Research, Christian Albrechts University, Kiel, Germany (KIA-) or the AMS ^{14}C Dating Centre at Aarhus University, Denmark (AAR-).

^The results have been calibrated using OxCal v 4.2.4 (Bronk Ramsey 2009) and the IntCal13 (Reimer et al. 2013) calibration data, with date ranges rounded outwards to the nearest 10 yr. The carbonized surface residue cannot be older than this date range. If the ^{14}C age is subject to a freshwater reservoir effect, the true date of the carbonized surface residue could be significantly more recent.

[#]Sherd numbers for Veksa 3 and Kääpa in Table 1 follow those used in Piezonka (2015); there are some differences between this publication and Piezonka (2008), in which some of the ^{14}C results discussed here were originally presented.

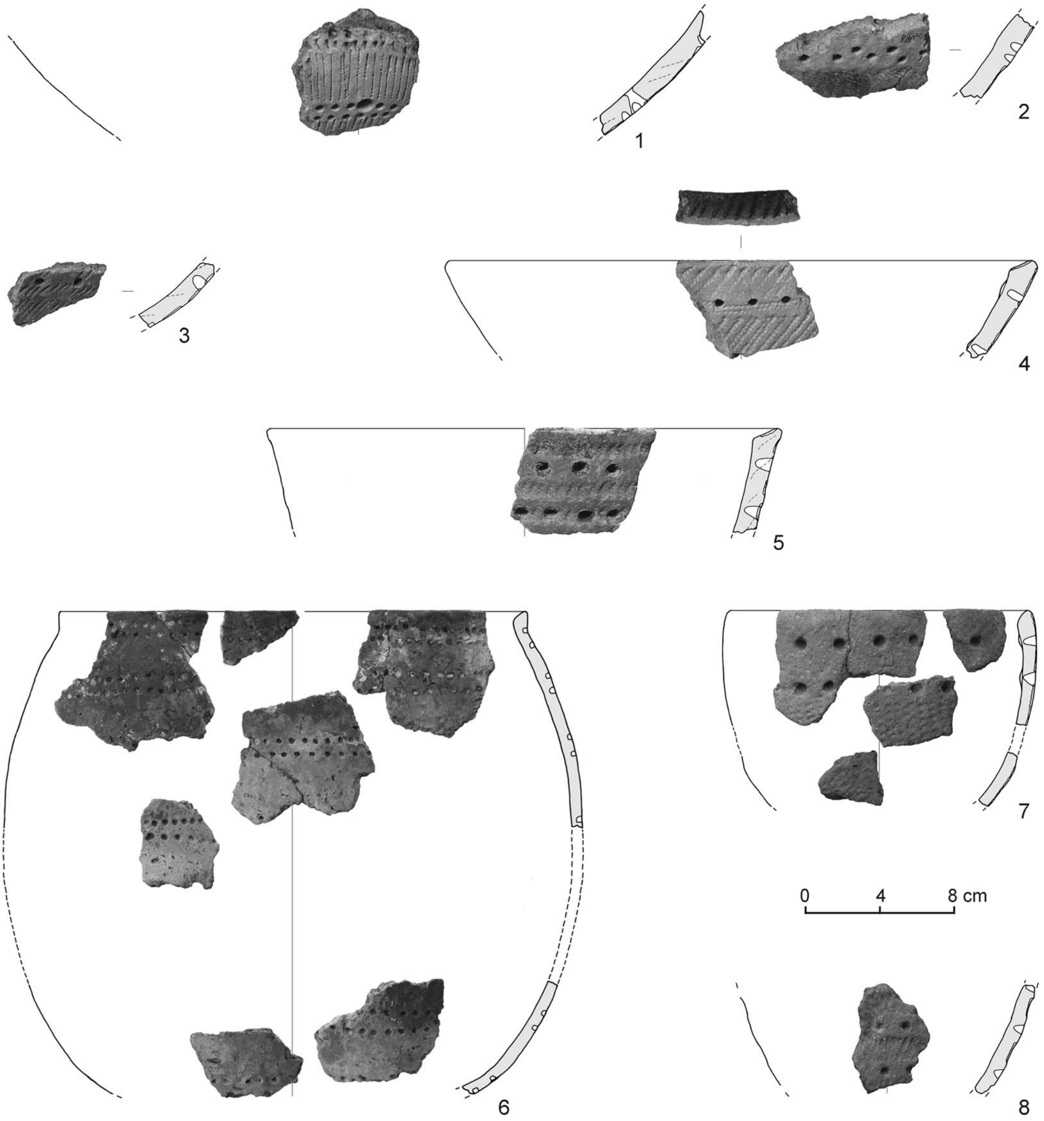

Figure 3 Veksa 3, Vologda province, Russia. Fragments of pottery from which organic residue samples were taken. 1 – sample KIA-49797, Upper Volga culture; 2 – sample KIA-49798, Earliest Comb-Pitted ware; 3 – sample KIA-49799, 2nd comb ceramic complex; 4 – sample KIA-33927, 2nd comb ceramic complex; 5 – sample KIA-33928, "Northern types"; 6 – samples KIA-33926 and KIA-49796, Narva; 7 – sample KIA-49790; 8 – sample KIA-49789, Comb-Pitted ware (illustration: H Piezonka).

Middle Neolithic Kargopol culture. Charred residue from a Kargopol potsherd was dated and analyzed (AAR-17169; Table 1).

Tudozero 5, Russia

Tudozero 5 is located at the southeastern bank of Lake Onega in the north of Vologda province (Figure 2). The stratified archaeological remains encompass evidence from the Mesolithic through to Medieval times. With regards to the introduction and development of Early Neolithic pottery in this region, the stratigraphic separation by a sterile layer of the earliest,

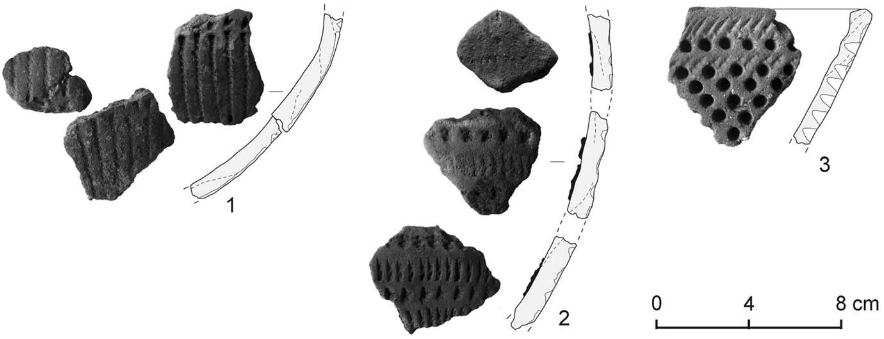

Figure 4 Karavaikha, Vologda province, Russia. Fragments of pottery from which organic residue samples were taken. 1 – Karavaikha 4, sample AAR-17172, Early Neolithic ware; 2 – Karavaikha 4, sample AAR-17171, Early Neolithic ware; 3 – Karavaikha 1, sample AAR-17169, Kargopol' (illustration: H Piezonka).

comb-decorated ware from an overlying layer with pottery resembling the Sperrings ware of Russian Karelia (Ivanishchev and Ivanishcheva 2000; Ivanishcheva et al. 2015) is especially important. Conventional ^{14}C dates from the associated cultural layers suggest a chronological position of the early Comb Ware complex in the second quarter of the 6th millennium cal BC and a time bracket for the Sperrings complex in the last quarter of the 6th and the beginning of the 5th millennium cal BC.[1] In this paper, AMS dating results and %C, %N, δ^{13}C, and δ^{15}N values are reported for foodcrust samples of a Comb Ware vessel from the lower layer (AAR-17174) and of a Sperrings vessel from the upper Early Neolithic layer (AAR-17173) (Table 1, Figure 5).

Sakhtysh 2a and Ozerki 17, Russia

Sakhtysh 2a and Ozerki 17 are among the well-investigated stratified peat-bog sites in the Upper Volga region (Figure 2). Their archaeological sequences start in the Mesolithic and cover several prehistoric periods. In a previous study, nine samples of organic residue adhering to Early Neolithic Upper Volga culture pottery from Sakhtysh 2a were AMS dated; from Ozerki 17, foodcrusts from one Upper Volga culture sherd and one Middle Neolithic Lyalovo culture sherd were dated (Hartz et al. 2012). Here, we report the results of the %C, %N, δ^{13}C, and δ^{15}N values measured in the dated samples (Table 1).

Kääpa, Estonia

The Stone Age settlement of Kääpa on the left bank of River Võhandu in southeastern Estonia (Figure 2) has yielded abundant archaeological finds including thousands of fragments of Early Neolithic Narva pottery (Jaanits 1968; Yanits 1976). The Early Neolithic complex is associated with a cultural horizon between the mineral subsoil and an overlying peaty layer, while later material of the Middle Neolithic Typical Comb Ware culture has been found at higher levels within the peat. Previous ^{14}C dates from Kääpa (Liiva et al. 1966) probably relate to the Typical Comb Ware phase, with the exception of one wild horse tooth dated to 4790–4550 cal BC (KIA-35737, 5820 ± 45 BP; Sommer et al. 2011). Of seven Narva vessels investigated here, two

---

[1] One conventional date (TA-2354, 7240 ± 60 BP), which according to Ivanishchev and Ivanishcheva (2000) also stems from the lower Early Neolithic horizon, appears unexpectedly old. It is not stated what material was dated; therefore, it cannot be judged whether an old-wood effect, a reservoir effect, a relocation from the Mesolithic complex at this site, or another external reason is responsible for the age offset.

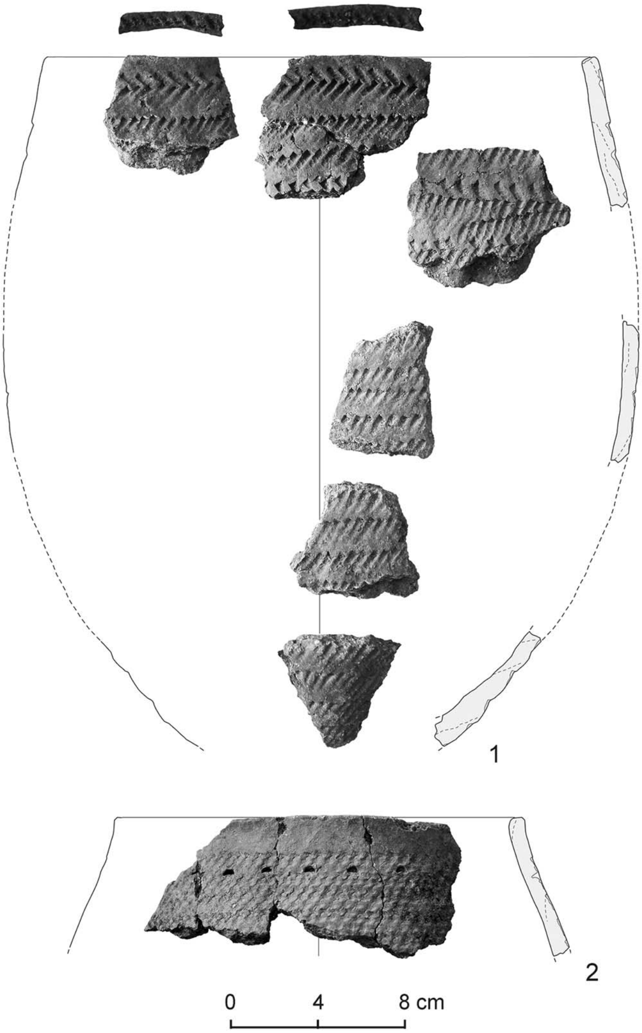

Figure 5 Tudozero 5, Vologda province, Russia. Fragments of pottery from which organic residue samples were taken. 1 – sample AAR-17174, Earliest Comb ware; 2 – sample AAR-17173, Sperrings (illustration: H Piezonka).

were dated previously and %C, %N, $\delta^{13}C$, and $\delta^{15}N$ have now been measured in the excess dating extracts (KIA-33921, KIA-35897; Piezonka 2008). Four sherds are dated here for the first time (KIA-49792, KIA-49793, KIA-49794, KIA-49795; Piezonka 2015), and EA-IRMS results are reported for one further sample of charred residue from a Narva lamp that was too small to date (Kä-2007/70; Piezonka 2015) (Table 1, Figure 6).

Radiocarbon Dating and Isotopic Analysis (AMS and EA-IRMS)

The samples listed in Table 1 were submitted for AMS ^{14}C dating to the Leibniz Laboratory for Radiometric Dating and Stable Isotope Research, Christian Albrechts University Kiel, Germany, in 2007 and 2013 (Ozerki 17, Sakhtysh 2a, Veksa 3, Kääpa), or to the AMS ^{14}C Dating Centre at Aarhus University, Denmark, in 2013 (Tudozero 5, Karavaikha 1, 4).

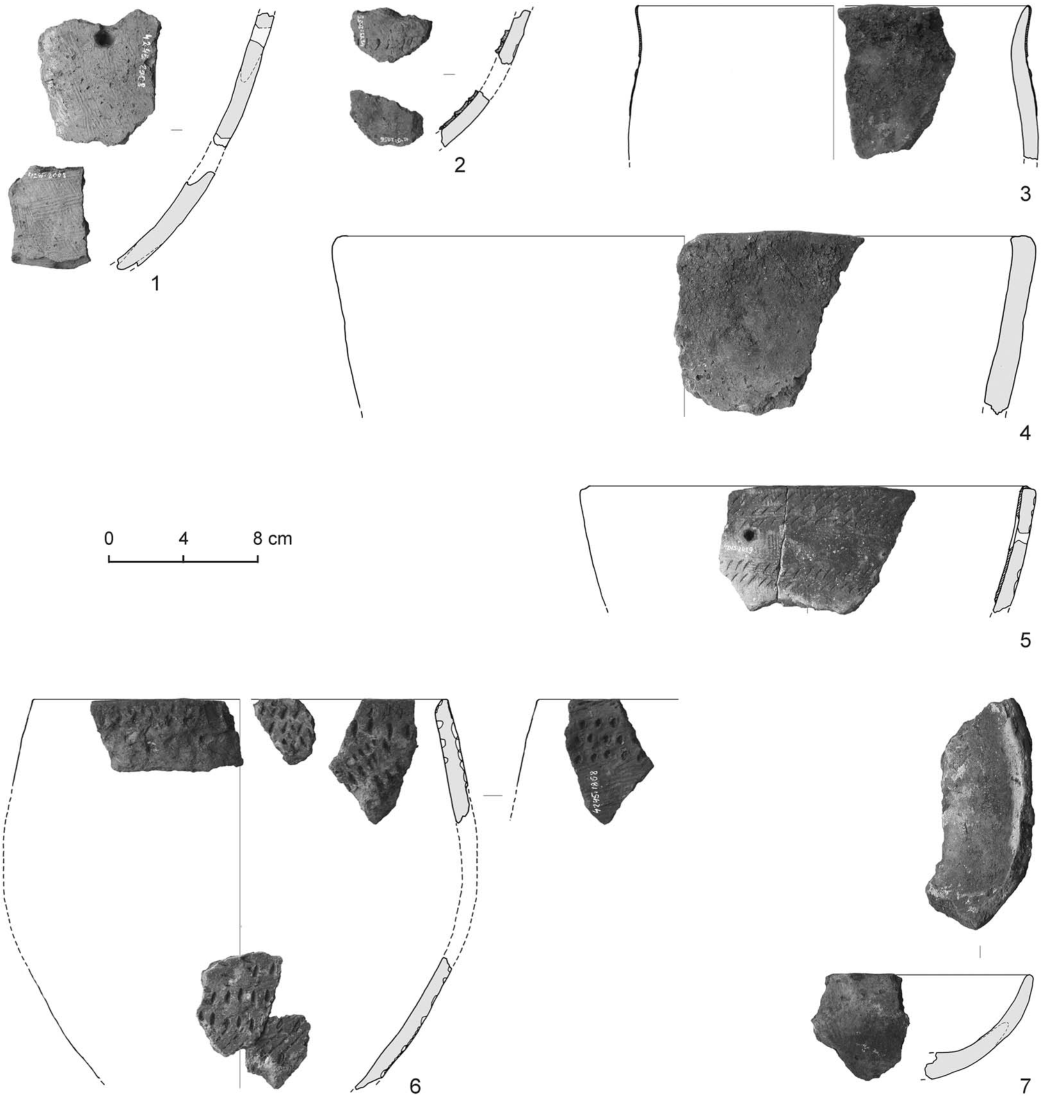

Figure 6 Kääpa, Estonia. Fragments of Narva pottery from which organic residue samples were taken. 1 – sample KIA-35897; 2 – KIA-49794; 3 – sample KIA-49795; 4 – sample KIA-49793; 5 – sample KIA-33921; 6 – sample KIA-49792; 7 – sample KIA-49791 (illustration: H Piezonka).

One sample (KIA-33929) consisted of sediment from a pit, containing bone (including small fragments of fish bone), burnt bone, and charcoal fragments; part of the charcoal was selected for dating. Aside from AAR-17170 (bone dagger), AAR-21042 (perforated elk tooth), and KIA-39300 (plant fiber), the other samples discussed here were all identified as carbonized surface residues adhering to the inner or outer surfaces of typologically diagnostic pottery (see Table 1 for details), and ~50 mg of material was selected. Carbonized surface residues from the inner and outer surfaces of one vessel were sampled separately (KIA-33926 and KIA-49796).

The Sakhtysh 2a and Ozerki 17 samples were initially treated with a sequence of solvents (Bruhn et al. 2001) to remove lipids. The subsequent chemical pretreatment for all samples, in both Kiel and Aarhus, was the conventional acid-base-acid (ABA) treatment (1% or 1M HCl at 60°C or 80°C for an hour, 1% or 0.5M NaOH at 60°C or 80°C for an hour, and again 1% or 1M HCl at room temperature overnight), in order to remove secondary carbonate and mobile organic components. The insoluble residue was then dried and weighed (Table 1; "yield" is expressed as a percentage of the starting weight of pretreated material). Of samples processed in Kiel, most produced extracts above 60% of the starting weight, but the charcoal sample KIA-33929 and foodcrust samples KIA-49789 and KIA-49790 gave poor yields (16–18%). There was insufficient extract from these samples for both stable isotope and AMS analysis.

We intentionally analyzed by EA-IRMS aliquots of the same chemical fraction of each foodcrust used for ^{14}C dating, in order to be able to comment on the likely origin of the carbon in the AMS targets. Samples from Veksa 3 and Kääpa were measured at the School of Life Sciences, University of Bradford, in duplicate on a Thermo Flash 1112 elemental analyzer coupled to a Thermo Delta plus XL mass spectrometer, to measure %C and %N contents, and ^{13}C/^{12}C and ^{15}N/^{14}N ratios (δ^{13}C and δ^{15}N). Samples from Ozerki 17 and Sakhtysh 2a were measured at the Berlin Natural History Museum, using a Thermo/Finnigan MAT V isotope ratio mass spectrometer, coupled to a Thermo Flash EA 1112 elemental analyzer. Samples from Karavaikha 1 and 4 and Tudozero 5 were analyzed at the AMS ^{14}C Dating Centre at Aarhus University, by combustion in a EuroVector elemental analyzer coupled to an IsoPrime stable isotope ratio mass spectrometer. Typical measurement errors of better than ±0.2‰ are quoted for δ^{13}C and δ^{15}N in samples and standards. Atomic C/N ratios were calculated from the elemental concentrations (Table 1).

RESULTS AND DISCUSSION

Vessel Use or Dietary Changes: Implications from EA-IRMS Results

Figure 7 shows the elemental concentrations in foodcrust extracts. The high %N values, and correspondingly low C/N values in most foodcrusts, are typical of extracted foodcrusts from hunter-gatherer-fisher pottery in the Baltic region (Philippsen 2013a; Philippsen and Meadows 2014; J Meadows, unpublished data). Figure 8 shows the foodcrust stable isotope ratios. The lowest δ^{13}C values are associated with the highest δ^{15}N values and low C/N ratios, and the lowest δ^{15}N values with moderate δ^{13}C values and the highest C/N ratios. A small group of samples from Sakhtysh 2a has low C/N ratios, moderate δ^{15}N values, and the highest δ^{13}C values.

Stable isotope values in prehistoric fauna and flora from the Vologda region where Veksa 3 is located are unknown, but we can infer from collagen δ^{13}C and δ^{15}N values in animal bones from the prehistoric burial ground at Minino, on Lake Kubena (Wood et al. 2013), ~50 km northwest of Veksa 3, that δ^{13}C values in plants and meat from terrestrial herbivores were quite restricted, as we would expect, given that native vegetation uses only the C_3 photosynthetic pathway (Figure 9). An elk tooth from an Early Metal Age burial at Sakhtysh 2a backs up this picture (Piezonka et al. 2013) (Table 1). Equally, there is no reason to suspect that the conventional model of δ^{15}N enrichment according to trophic level is invalid in this region, although, due to the northern climate, the baseline soil δ^{15}N may perhaps be lower than in central and southern Europe (Amundson et al. 2003). Three fish bones and one aquatic bird bone from Minino indicate that freshwater resources were relatively depleted in δ^{13}C and enriched in δ^{15}N, but it is particularly the range of human bone δ^{13}C and δ^{15}N values that shows that most fish in this catchment must have had much lower δ^{13}C and much higher δ^{15}N values than terrestrial foodstuffs. The pattern is similar (for example) to that seen at Riņņukalns

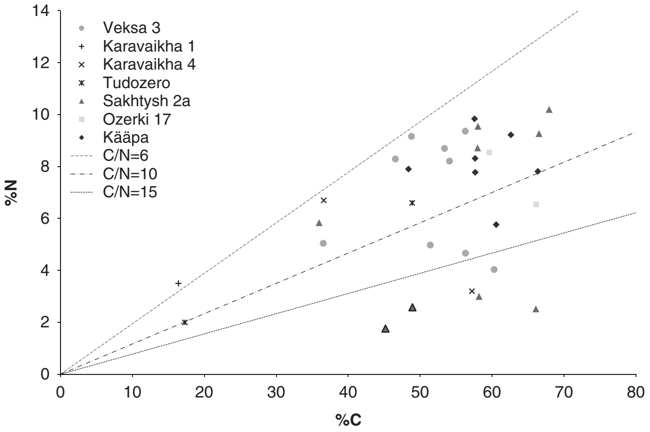

Figure 7 Elemental concentrations (by % weight). With the exception of the two outlined triangles (KIA-39311-39312: acid-insoluble, alkali-soluble residues), all foodcrust extracts analyzed were acid- and alkali-insoluble residues. The dashed lines represent atomic C/N ratios; most samples fall between C/N = 6 and C/N = 10, regardless of differences in %C and %N (graph: J Meadows).

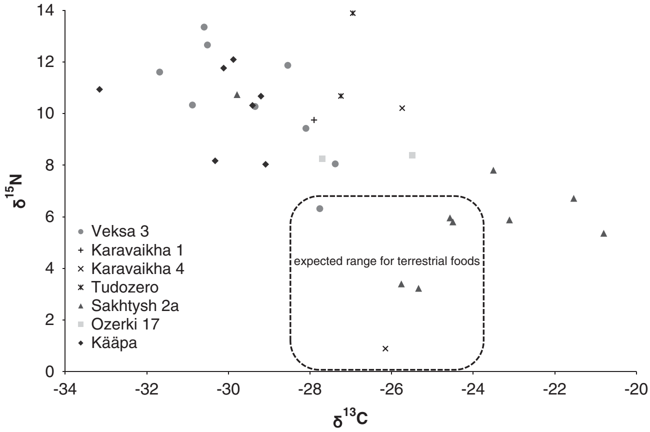

Figure 8 Bulk foodcrust stable isotope results (see Table 1). The expected range for terrestrial foods (plants and animal flesh) is based on the herbivore collagen data shown in Figure 9 (graph: J Meadows).

in the Lake Burtnieks region of Latvia, where we have much more isotope data for fish (Figure 9). Although the Minino material covers a wide date range, the two human individuals directly dated to the Early Neolithic period (M1 4 and 13) have among the lowest $\delta^{13}C$ and the highest $\delta^{15}N$ values, confirming that the isotopic differences between aquatic and terrestrial species apply during the period of interest at Veksa 3. Two Middle Neolithic and two Early Metal Age human individuals from Sakhtysh 2a in the Upper Volga region have produced comparable isotopic values, which are seen as indicating a diet rich in aquatic resources (Figure 9; Piezonka et al. 2013).

Foodstuffs consist mainly of carbohydrates, fats, proteins, and (in the case of plants) fiber. Fats and carbohydrates are nitrogen-free, and only protein is nitrogen-rich. Thus, a food with high

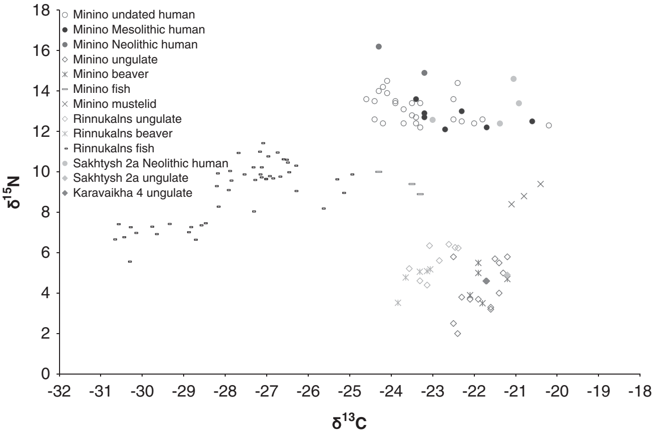

Figure 9 Collagen stable isotope results from early- to mid-Holocene fauna and human remains from Minino, Vologda province, Russia (Wood et al. 2013); Karavaikha 4, Vologda province, Russia (this paper); Sakhtysh 2a, Ivanovo province, Russia (Piezonka et al. 2013 and this paper); and fauna from Riņņukalns, Latvia (Bērziņš et al. 2014; Meadows et al. 2016; Schmölcke et al. 2016) (graph: J Meadows).

fat or carbohydrate content should have a higher C/N value than one that is rich in protein. In theory, foodcrust δ^{15}N values will be determined by the ingredients with the highest protein contents, but δ^{13}C values (and ^{14}C ages) may also reflect the carbon content of sugary, starchy, or fatty ingredients, which may not be from the same organisms as proteins. Even within a single organism, lipids have significantly lower δ^{13}C values than proteins. However, low C/N values (Figure 7) suggest that high-protein ingredients predominated in most of our foodcrusts, and we may therefore use δ^{15}N and δ^{13}C values in herbivore collagen (Figure 9) to estimate the relevant isotopic range for terrestrial foods (Figure 8).[2] An important distinction between interpreting stable isotope data from foodcrusts and from human bones is that whereas the *average* isotope values of different food groups are relevant to human bone collagen, the *range* of values is more pertinent to foodcrusts. Such variability may reflect not only the complexity of food webs but also factors such as seasonality (which may affect e.g. fat content), the number of cooking events incorporated in individual crusts, etc.

A further complication in the interpretation of the stable isotope data from bulk foodcrusts is that some fractionation is possible, both during cooking and charring, and perhaps during burial. Experimental work with fish (Fernandes et al. 2014), cereals (e.g. Fraser et al. 2013), and artificial foodcrust (Philippsen 2013b) does not suggest large isotopic shifts during cooking and charring, but there are few data for diagenesis (Heron and Craig 2015). When comparing samples from the same burial environment, we may argue that it is unlikely that differences in diagenesis would create coherent patterns in the stable isotope results, although we should be wary of overinterpreting the results from individual samples. More specific information on food and non-food products in pottery vessels can be provided by biomolecular analysis of

---

[2] Herbivore collagen is typically δ^{13}C enriched by 5‰ and δ^{15}N-enriched by 3–4‰ compared to plant foods (e.g. DeNiro and Epstein 1981; Lee-Thorp et al. 1989), while bulk flesh is typically slightly δ^{15}N-enriched and δ^{13}C-depleted by 2–3‰ compared to collagen of the same animal (e.g. Fischer et al. 2007). Thus, herbivore collagen values (δ^{13}C –23 to –21‰, δ^{15}N 4 to 6‰) suggest ranges of –28 to –24‰ δ^{13}C and 0–7‰ δ^{15}N for terrestrial foods, before any fractionation due to charring and diagenesis.

foodcrusts and/or organic residues within the pottery matrix, when preservation conditions favor the survival of characteristic molecules known as biomarkers. Gas chromatography (GC), gas chromatography mass spectrometry (GCMS), and/or gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS) allow lipids in particular to be attributed to oils, waxes, and fats from terrestrial plants, terrestrial animals, marine mammals, and fish (Craig et al. 2007, 2011; Evershed 2008; Heron and Craig 2015). These techniques, however, do not quantify the contribution of the specific constituents to the overall carbon content.[3]

Notwithstanding the aforementioned qualifications, stable isotope values from almost all the Kääpa, Veksa 3, Ozerki, Karavaikha, and Tudozero foodcrusts (Table 1, Figure 8) are more consistent with freshwater species than with the meat of terrestrial herbivores, as $\delta^{13}C$ values are depleted while $\delta^{15}N$ values are enriched. The same pattern in foodcrusts on Ertebølle pottery from inland sites in Schleswig-Holstein is associated with what appear to be large FREs (Philippsen and Meadows 2014), and it is therefore sensible to regard the calibrated foodcrust dates reported in Table 1 as *termini post quos* for the dates of the pots.

The Sakhtysh 2a results do not fit the general pattern, as only one sample (KIA-39310) is depleted in $\delta^{13}C$ and enriched in $\delta^{15}N$, but this is the same sample that Hartz et al. (2012) identified as having an unacceptably high ^{14}C age, and thus (most probably) a significant FRE. The only foodcrust ^{14}C age that we can confidently say is not subject to a significant FRE is KIA-39301 (also from Sakhtysh 2a), as it is consistent with KIA-39300, the ^{14}C age of a willow string embedded in the foodcrust of the same pot (Figure 10). The EA-IRMS results from KIA-39301 (Table 1) are entirely consistent with this outcome: the low $\delta^{15}N$ and moderate $\delta^{13}C$ place this sample within the expected range for terrestrial foods (Figure 8), and the high C/N value suggests that plant ingredients may have been important (Yoshida et al. 2013). Three Sakhtysh 2a samples (KIA-39308, -39309, and -39311) are unusual in the overall scheme, having relatively enriched $\delta^{13}C$, moderate $\delta^{15}N$, and low C/N values. Their ^{14}C ages are the earliest for Upper Volga pottery deemed acceptable by Hartz et al. (2012).

We see an interesting trend in the EA-IRMS results: isotope values appear to become more aquatic over time (Figure 8), even within the Early Neolithic. At Veksa 3, for example, samples from the oldest pottery types (KIA-49797, Upper Volga culture and KIA-49798, "Earliest Comb-Pitted Ware") are least depleted in $\delta^{13}C$ and have at the same time the lowest $\delta^{15}N$ values, and among the highest C/N values (Figure 11). The slightly younger "2nd Comb Ware complex" samples (KIA-33927 and KIA-49799) have lower $\delta^{13}C$ values and are more enriched in $\delta^{15}N$. The sample from the "Northern Types" vessel (KIA-33928) is even more depleted in $\delta^{13}C$. The two samples taken from the Narva-type vessel (KIA-33926 and KIA-49796) have some of the highest $\delta^{15}N$ values in the series, and the most "fishy" EA-IRMS results come from the two Comb-Pitted Ware pots found in layer 6, which unfortunately could not be dated due to the low carbon contents (KIA-49789 and KIA-49790). While the number of samples is still too small to draw firm conclusions, the isotopic data allow us to further advance a hypothesis put forward by Hartz et al. (2012) that in the northeast European forest zone the intensity of processing aquatic products in ceramic containers increased gradually in the 6th and 5th millennia cal BC. Interestingly, the Mesolithic and Early Neolithic human bone samples

---

[3] Results of biomolecular analyses at the University of York of foodcrusts and organic residues in the pottery matrix of prehistoric ceramic vessels from inland sites in the Vologda region of the Russian forest zone, spanning the period from the first introduction of pottery into the region in the early 6th millennium cal BC through to the Early Iron Age in the 1st millennium cal BC, will be reported in a subsequent paper.

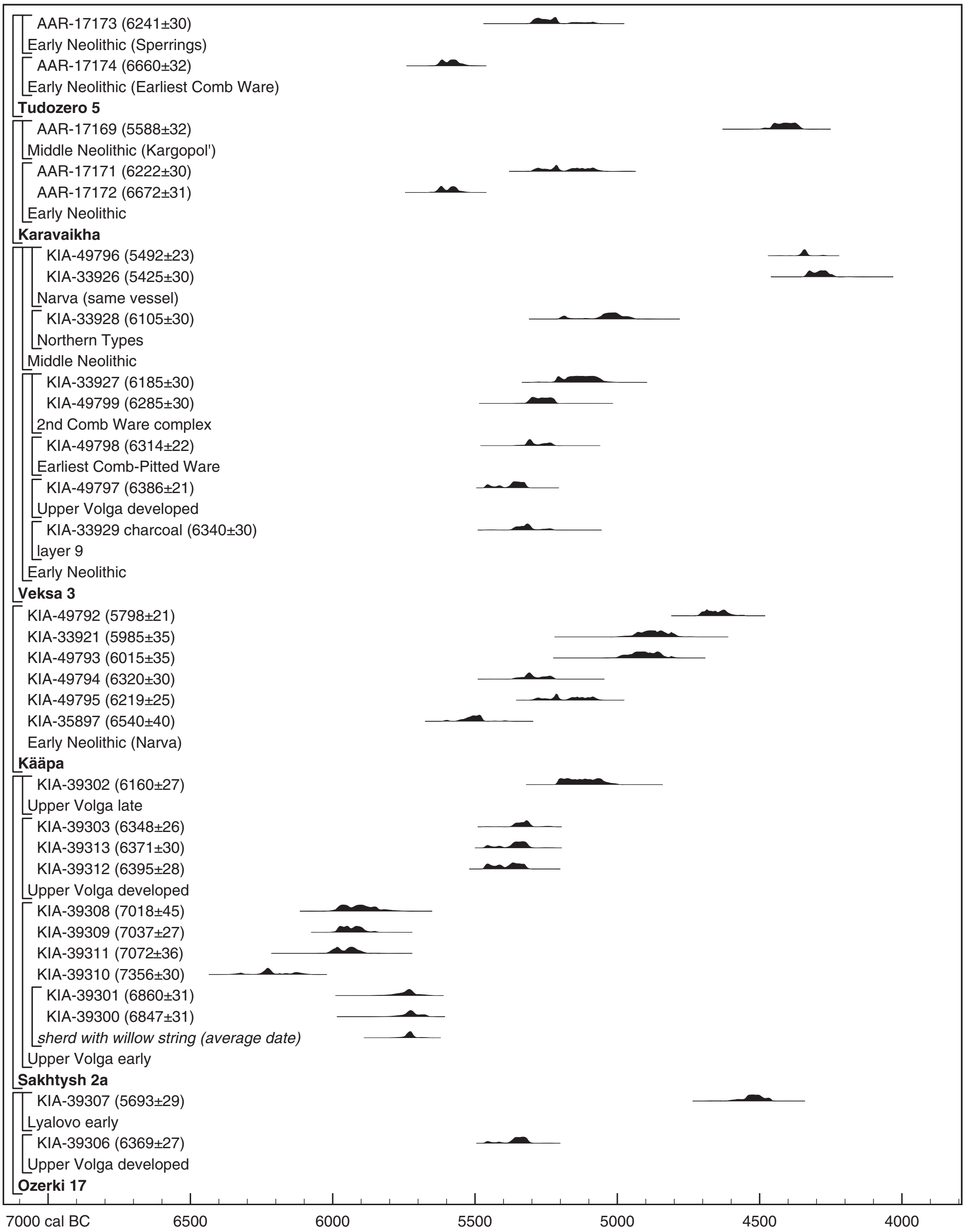

Figure 10 Calibration of ^{14}C results (reported in Table 1) using OxCal v 4.2.4 (Bronk Ramsey 2009) and the IntCal13 data (Reimer et al. 2013) (graph: J Meadows).

from Minino show a similar pattern, of becoming more depleted δ^{13}C and more enriched δ^{15}N over time, which is seen as a possible sign of an increase in the consumption of freshwater fish (Wood et al. 2013:173–4). Other possible explanations, such as climatic and ecological developments or changes in the exploitation strategies of natural resources, must also be taken

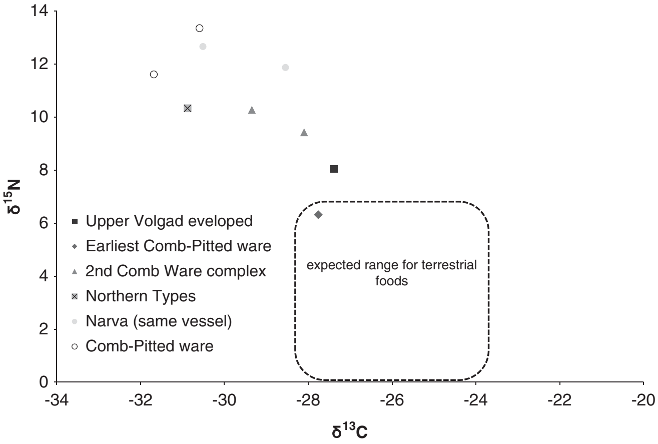

Figure 11 Stable isotope results from Veksa 3 foodcrusts (Table 1). Symbols represent the typological sequence from Upper Volga (earliest) to Comb-Pitted wares (latest) (graph: J Meadows).

into account, and further research into the isotopic values of natural resources from the period in question is needed to better understand the mechanisms that lie behind the observed pattern.

Stratigraphy, Pottery Typology, and Absolute Chronology

As the presence of carbon from aquatic species can only produce older dates, a gradual increase over time in the use of aquatic resources could lead to spurious "reversals" in the ^{14}C ages of foodcrusts, i.e. could make foodcrusts on more recent pottery appear to be older than foodcrusts on earlier sherds. Most results we have are consistent with stratigraphy and the expected typological sequences, however (Figure 10). The number of samples dated is small relative to the period of time spanned by this study, and it would be easy to overlook moderate FREs, particularly if most samples were affected to some degree, but altogether there is surprisingly little evidence of large FREs in foodcrust dates.

We have no direct evidence yet that there was a significant FRE at Veksa 3. The evidence from nearby Minino (Wood et al. 2013, see discussion above), however, implies large FRE offsets in fish. Thus, the ^{14}C ages of foodcrusts from this region in which fish was a major source of carbon should be several centuries too old. Nevertheless, the relative sequence at Veksa 3 suggested by the ^{14}C results is in accordance with the stratigraphic and typological information (Figures 3, 7; see Nedomolkina 2004; Piezonka 2015:43–5). KIA-49797 (6386 ± 21 BP, apparently the oldest date on foodcrust) is from a vessel of the developed Upper Volga culture, a type that is associated mainly with the upper part of cultural layer 9 and the lower part of layer 8 above. In addition to KIA-33929 (6340 ± 30 BP), from charcoal in layer 9, six conventional ^{14}C dates reported to stem from layers 9/8 range between 6950 ± 150 BP (Le-5866) and 6220 ± 150 BP (Le-5868) (Figure 12; Timofeev et al. 2004; Piezonka 2008). Although detailed information on context or dated material is not available, these results support the idea that any FRE in KIA-49797 is probably negligible. KIA-49798 (6314 ± 22BP) is from a sherd of a rare type preliminarily named "Earliest Comb-Pitted Ware" at Veksa 3 that is concentrated in the horizon between the upper part of layer 9 and the lower part of layer 8. KIA-49799 (6285 ± 30 BP) and KIA-33927 (6185 ± 30 BP) both belong to the so-called "2nd Comb Ware complex," which is mainly found in the upper part of cultural layer 8. The next date in the sequence, KIA-33928 (6105 ± 30 BP) comes from a vessel associated with the "Northern Types" pottery that is mainly

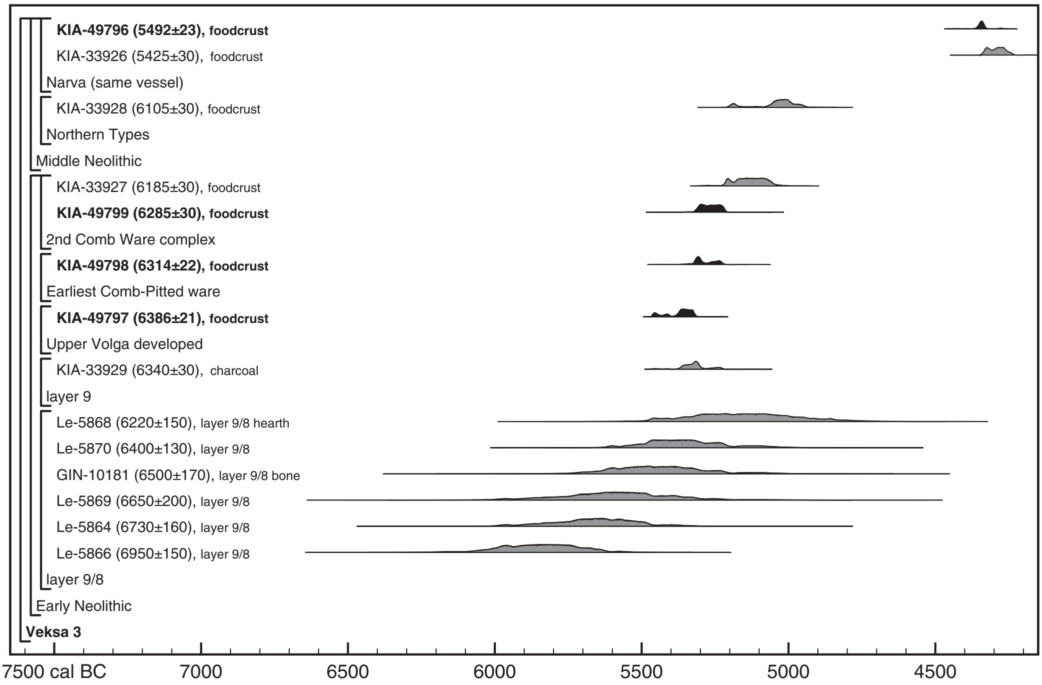

Figure 12 Calibration of ^{14}C dates from the Neolithic layers at Veksa 3. Black: new results (this paper, Table 1); gray: previously published dates (Timofeev et al. 2004; Piezonka 2008) (graph: J Meadows).

found in layer 7. Finally, two statistically consistent results (KIA-33926, 5425 ± 30 BP, and KIA-49796, 5492 ± 23BP) were obtained on the internal and external foodcrusts of a vessel that is typologically comparable to ceramics of the eastern Baltic Narva culture of the second half of the 5th millennium cal BC and thus is also well in accordance with the expected age (Piezonka 2008, 2015:48).

At Karavaikha 4, 14 ^{14}C dates from the lower cultural horizon, most of them on wooden artifacts, span from 7050 ± 80 BP (SPb-1300) to 6030 ± 130 BP (GIN-12514), contradicting the assumption that this horizon represents a confined Early Neolithic episode of human activity (Figure 13). The date of the bone dagger (AAR-17170, 7009 ± 40 BP) is among the oldest (Table 1), indicating human presence at the site in the first third of the 6th millennium cal BC, a period associated with the aceramic Late Mesolithic in these parts of northern European Russia (Filatova 2006). The earliest date directly associated with pottery stems from the foodcrust of vessel 5 (AAR-17172, 6672 ± 31 BP), which is typologically similar to pottery of the second phase of the Upper Volga culture. This date forms a group with four broadly contemporary conventional dates from wood samples. Compared to dates for the developed phase of Upper Volga pottery elsewhere, however, the date from Karavaikha 4 seems too early. Foodcrust dates for typologically connected wares and their contexts from Veksa 3, Sakhtysh 2a, and Ozerki 17 (Hartz et al. 2012) are ~400 ^{14}C yr younger (Figure 10). A significant FRE could therefore have affected AAR-17172. The second foodcrust date from Karavaikha 4 (AAR-17171, 6222 ± 30 BP) is the second-youngest date associated with the lower cultural horizon. Its typological attribution is not as straightforward as with other sherds discussed here. While the composition of the decoration stylistically resembles the “Northern Types,” the use of irregular stamps instead of large, deep pits is an atypical feature. The dating result appears

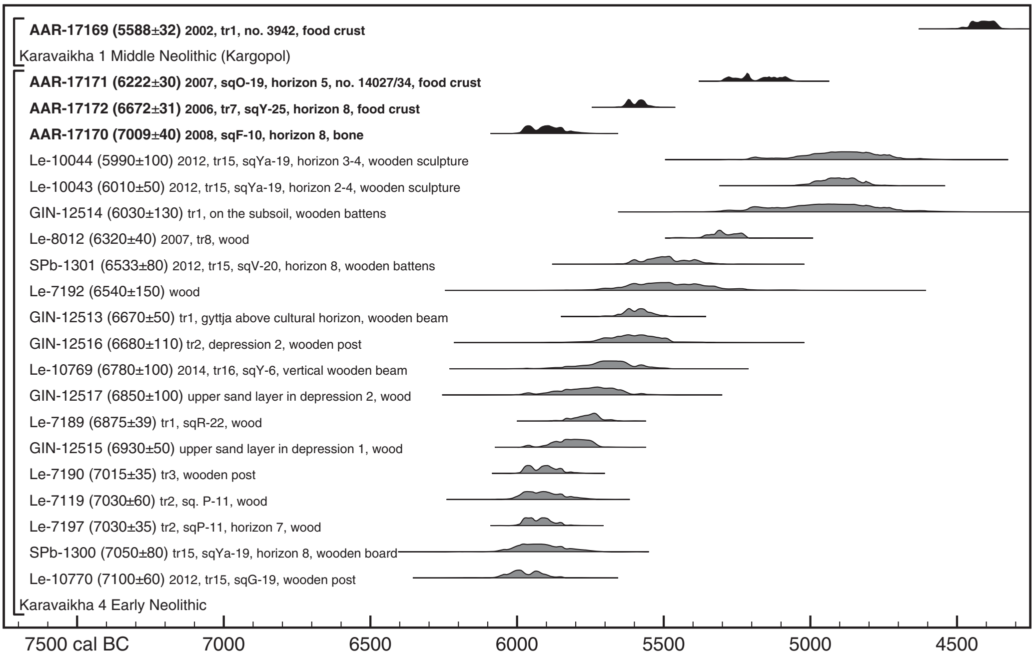

Figure 13 Calibration of ^{14}C dates from Karavaikha 1 and 4. Black: new results (this paper, Table 1); gray: previously published dates and unpublished conventional dates (Kosorukova 2007; Kiryanova and Kosorukova 2013; this paper) (graph: J Meadows).

marginally older than that for "Northern Types" pottery from Veksa 3 (KIA-33928). There are no other dates for contexts with this type of pottery at Veksa 3 or in the Upper Volga region. Altogether, the chronology of the lower cultural horizon at Karavaikha 4 is not fully understood, and it seems likely that several phases of activity in the Late Mesolithic and Early Neolithic are represented. To understand the chronological setting of the pottery associated with it and to judge the possible presence of FRE in its foodcrusts, dating of securely associated terrestrial material (e.g. plant fibers and resins used to repair broken pots) will be necessary. The ^{14}C age of the foodcrust on the Kargopol culture sherd from Karavaikha 1 (AAR-17169, 5588 ± 32 BP) is in broad accordance with its expected position in the early phase of this Middle Neolithic culture (Piezonka 2015), although the contextual and typological information on chronology is in this case not detailed enough to decide whether a FRE might have affected the date or not.

The two foodcrust dates from Tudozero 5 fit both the stratigraphic sequence and existing conventional ^{14}C dates from the respective layers (Figure 14). They thus confirm the assumption that the local early Comb Ware is associated with an Early Neolithic horizon dating to the second quarter of the 6th millennium cal BC, while Sperrings pottery belongs to a later phase of the Early Neolithic in the last third of the 6th millennium cal BC. Altogether, stratigraphic and typological evidence and associated ^{14}C dates suggest that no substantial FRE has affected the foodcrust dates from Tudozero 5. At the same time, the EA-IRMS results and especially the high δ^{15}N values suggest a significant aquatic component.

The chronological implications of the AMS dates on pottery crusts from Sakhtysh 2a and Ozerki 17 have been discussed elsewhere (Hartz et al. 2012). Here, we stress again the

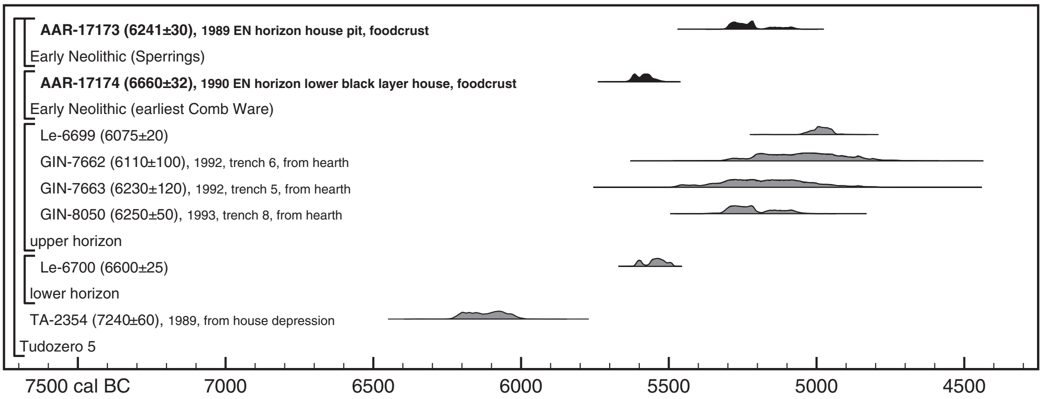

Figure 14 Calibration of ^{14}C dates from the Early Neolithic layers at Tudozero 5. Black: new AMS results (this paper, Table 1); gray: previously published dates (Ivanishchev and Ivanishcheva 2000) (graph: J Meadows).

significance of the very good correlation of the expected presence/absence of FRE and of the isotopic signals of some samples at Sakhtysh 2a.

Foodcrusts on typologically more-or-less uniform Narva pottery from the Early Neolithic cultural horizon at the Estonian site of Kääpa have ^{14}C ages between 6540 ± 40 BP (KIA-35897) and 5798 ± 21 BP (KIA-49792) (Figure 10), representing offsets of between zero and 720 ± 60 ^{14}C yr relative to the date of the horse tooth (KIA-35737, 5820 ± 45 BP; Sommer et al. 2011). All EA-IRMS data from the dated foodcrusts suggest a high freshwater aquatic component, but there is no correlation between foodcrust ^{14}C ages and EA-IRMS results that might be used to estimate FREs. Highly variable FREs were recorded in studies of modern freshwater fish in Ireland and Germany (Keaveney and Reimer 2012; Philippsen 2013a) and in recent lake sediment upstream of Kääpa (Alliksaar and Heinsalu 2012). As foodcrusts may represent single cooking episodes, such variability might account for the scatter of foodcrust ^{14}C ages at Kääpa, but we cannot assume that the dated sherds are contemporaneous with each other or the horse tooth. Without more dates on terrestrial material from the Early Neolithic complex, we cannot decide at the moment whether, and if so, to what extent, the dates have been affected by FRE.

CONCLUSIONS AND PERSPECTIVES

Our EA-IRMS results, in the context of stable isotope and ^{14}C data from Minino and other prehistoric sites in the northeastern forest zone, serve to emphasize that a significant proportion of the carbon in many (if not most) Stone Age foodcrust samples in this region is likely to have been derived from aquatic resources. While it appears that both δ^{13}C and δ^{15}N values are useful indicators of the presence of freshwater fish, much more detail about their variability in local terrestrial and aquatic food chains (as well as a larger number of foodcrust and human bone samples) would be required to confirm our impression that reliance on aquatic resources increased in the course of the Neolithic. If valid, however, this trend would imply that ^{14}C dates for the earliest pottery in this region would generally be the least affected by FRE.

The magnitude of FRE in individual foodcrusts is almost impossible to estimate without independent evidence of the absolute dates of pot sherds concerned, particularly in complex freshwater systems where the variability in FRE in aquatic species is unknown. At Veksa 3, we can infer the chronological sequence of the sherds, based on typological development and stratigraphy, but the only AMS ^{14}C date from a terrestrial sample so far simply provides an upper age limit for all the sherds. Without a lower age limit, or *terminus ante quem*, it is impossible to exclude large FREs, even in the Early Neolithic foodcrusts.

Nevertheless, the experimental approach followed by Philippsen et al. (2010) to better understand the relationship between foodcrust ingredients and dating results is promising, suggesting that EA-IRMS of foodcrusts can identify those most likely to be subject to FREs, provided that isotopic values in the local food ingredients are sufficiently well known and distinctive. Two foodcrust samples from Sakhtysh 2a clearly support this approach: KIA-39300 gave EA-IRMS results consistent with mainly plant ingredients, and a ^{14}C age fitting that from a plant fiber in the same vessel, whereas KIA-39310 produced an implausibly old ^{14}C age and EA-IRMS results suggesting that fish was the main ingredient. Three other samples from Sakhtysh 2a gave relatively high ^{14}C ages and EA-IRMS results that are difficult to interpret, and do not correspond to those from other sites.

Future research should therefore focus on measuring the range of isotopic values in relevant materials (bones of terrestrial animals, fish bones, mussel shells, plant remains, etc.) from the same region and period or, if possible, even from the same context as the foodcrust samples. Paired dates of foodcrusts and terrestrial material associated with the same vessel (e.g. Piličiauskas and Heron 2015) can also help to shed more light on FREs and their relation to EA-IRMS data, and paired human-herbivore (or plant) ^{14}C samples from closed contexts in the same region and period also provide useful information about the scale of local FREs. To understand the potential variability in foodcrust FREs, however, we also need to date multiple fish remains from closely dated contexts. At Veksa 3 and Sakhtysh 2a, new fieldwork will address these questions in the near future.

ACKNOWLEDGMENTS

EA-IRMS measurements and the 2013 AMS dates for Veksa 3 and Kääpa were funded by the Center for Baltic and Scandinavian Archaeology, Foundation of the Schleswig-Holstein State Museums, Schloss Gottorf, as part of the Man and Environment research theme. This paper has benefited substantially from discussions with Dr Oliver Craig, University of York, and the comments of three anonymous reviewers.

REFERENCES

Alliksaar T, Heinsalu A. 2012. A radical shift from soft-water to hard-water lake: palaeolimnological evidence from Lake Kooraste Kõverjärv, southern Estonia. *Estonian Journal of Earth Sciences* 61(4):317–27.

Amundson R, Austin AT, Schuur EAG, Yoo K, Matzek V, Kendall C, Uebersax A, Brenner D, Baisden WT. 2003. Global patterns of the isotopic composition of soil and plant nitrogen. *Global Biogeochemical Cycles* 17(1):1031.

Bērziņš V, Brinker U, Klein C, Lübke H, Meadows J, Rudzīte M, Schmölcke U, Stümpel H, Zagorska I. 2014. New research on Riņņukalns, a Neolithic freshwater shell midden in northern Latvia. *Antiquity* 88(341):715–32.

Bronk Ramsey C. 2009. Bayesian analysis of radiocarbon dates. *Radiocarbon* 51(1):337–60.

Bruhn F, Duhr A, Grootes PM, Mintrop A, Nadeau M-J. 2001. Chemical removal of conservation substances by 'Soxhlet'-type extraction. *Radiocarbon* 43(2A):229–37.

Craig OE, Forster M, Andersen SH, Koch E, Crombé P, Milner NJ, Stern B, Bailey GN, Heron CP. 2007. Molecular and isotopic demonstration of the processing of aquatic products in northern European prehistoric pottery. *Archaeometry* 49(1):135–52.

Craig OE, Steele VJ, Fischer A, Hartz S, Andersen SH, Donohoe P, Glykou A, Saula H, Jones DM, Koch E, Heron CP. 2011. Ancient lipids reveal continuity in culinary practices across the transition to agriculture in Northern Europe. *Proceedings of the National Academy of Sciences of the USA* 108(44):17,910–5.

DeNiro MJ, Epstein S. 1981. Influence of diet on the distribution of nitrogen isotopes in animals. *Geochimica et Cosmochimica Acta* 45(3):341–51.

Evershed RP. 2008. Organic residue analysis in archaeology: the archaeological biomarker revolution. *Archaeometry* 50(6):895–924.

Fernandes R, Meadows J, Dreves A, Nadeau M-J, Grootes P. 2014. A preliminary study on the influence of cooking on the C and N isotopic composition of multiple organic fractions of fish (mackerel and haddock). *Journal of Archaeological Science* 50:153–9.

Filatova VF. 2006. Voprosy proiskhozhdenija i etnokulturnoi prinadlezhnosti naseleniya epokhi mezolita (Questions of the emergence and ethnocultural position of the population of the Mesolithic period). In: Kochkurkina SI, Kosmenko MG, editors. *Problemy etnokulturnoi istorii naseleniya Karelii (mezolit-srednevekove)* (Problems of the ethno-cultural history of the Population of Karelia). Petrozavodsk. p 14–72.

Fischer A, Olsen J, Richards M, Heinemeier J, Sveinbjörnsdóttir ÁE, Bennike P. 2007. Coast–inland mobility and diet in the Danish Mesolithic and Neolithic: evidence from stable isotope values of humans and dogs. *Journal of Archaeological Science* 34(12):2125–50.

Fraser RA, Bogaard A, Charles M, Styring AK, Wallace M, Jones G, Ditchfield P, Heaton THE. 2013. Assessing natural variation and the effects of charring, burial and pretreatment on the stable carbon and nitrogen isotope values of archaeobotanical cereals and pulses. *Journal of Archaeological Science* 40(12):4754–66.

Hartz S, Lüth F, Terberger T, editors. 2011. Early pottery in the Baltic. Proceedings of the International Workshop 'Early Pottery in the Baltic,' Schleswig, 20–21 October 2006. *Bericht der Römisch-Germanischen Kommission* 89.

Hartz S, Kostyleva E, Piezonka H, Terberger T, Tsydenova N, Zhilin MG. 2012. Hunter-gatherer pottery and charred residue dating: new results on early ceramics in the north Eurasian forest zone. *Radiocarbon* 54(3–4):1033–48.

Heron C, Craig OE. 2015. Aquatic resources in foodcrusts: identification and implication. *Radiocarbon* 57(4):707–19.

Ivanishchev AM, Ivanishcheva MV. 2000. Tudozero-V poselenie pozdnego mezolita – rannego neolita v Yuzhnom Prionezh'e (Tudozero-V, a settlement of the late Mesolithic-early Neolithic in the south Onegy region). *Tverskoi Arkheologicheski Sbornik* 4(1):284–96.

Ivanishcheva MV, Kulkova MA, Sapelko TV. 2015. Natural processes in Holocene in the South Onega Lake region (on the basis of complex studies of multilayer settlement Tudozero V). In: Lozovskii VM, Lozovskaya OV, Vybornov AA, editors. *Neolithic Cultures of Eastern Europe: Chronology, Paleoecology and Cultural Traditions*. Materials of the international conference dedicated to the 75th anniversary of Viktor Petrovich Tretyakov, May 12–16, 2015, St Petersburg. St Petersburg: Izdatel'stvo OOO "Periferiya." p 285–9.

Jaanits L. 1968. Die frühneolithische Kultur in Estland. *Congressus Secundus Internationalis Fenno-Ugristarum, Helsinki 23.-28.8.1965, Pars 2*. Helsinki. p 12–25.

Jordan P, Zvelebil M. 2009. *Ex oriente lux*: the prehistory of hunter-gatherer ceramic dispersals. In: Jordan P, Zvelebil M, editors. *Ceramics Before Farming: The Dispersal of Pottery Among Prehistoric Eurasian Hunter-Gatherers*. Walnut Creek: Left Coast Press. p 33–89.

Karmanov VN, Zaretskaya NE, Volokitin AV. 2014. Another way of early pottery distribution in eastern Europe? Case study of the Pezmog 4 site, European far northeast. *Radiocarbon* 56(2):733–41.

Keaveney EM, Reimer PJ. 2012. Understanding the variability in freshwater radiocarbon reservoir offsets: a cautionary tale. *Journal of Archaeological Science* 39(5):1306–16.

Kiryanova AV, Kosorukova NV. 2013. K voprosu o datirovke torfyanikovoi stoyanki Karavaikha 4 v basseine ozera Vozhe [On the question of the dating of the peatbog site of Karavaikha 4 in Lake Vozhe basin]. In: *Geologo-Arkheologicheskie issledovaniya v timano-severouralskom regione*. Doklady 16-i nauchnoi konferencii 31 oktyabrya 2013g. Syktyvkar. p 105–11.

Kosorukova NV. 2007. Perekhod ot mezolita k neolitu v bassejne ozera Vozhe (novye materialy) [The transition from the Mesolithic to the Neolithic in the basin of Lake Vozhe (new materials)]. In: *Russkaya kultura novogo stoletiya: problemy izucheniya, sokhraneniya I ispolzovaniya istoriko-kulturnogo naslediya.* Sbornik statei. Vologda: Knizhnoe nasledie. p 41–52.

Lee-Thorp JA, Sealy JC, van der Merwe NJ. 1989. Stable carbon isotope ratio differences between bone collagen and bone apatite, and their relationship to diet. *Journal of Archaeological Science* 16(6):585–99.

Liiva A, Ilves E, Punning JM. 1966. Tartu radiocarbon dates I. *Radiocarbon* 8:434–41.

Lorenz S, Nedomolkina NG, Piezonka H. 2012. Geoarchaeology and floodplain development at the outstanding multiperiod dwelling site of Veksa 3 in the Sukhona basin. In: Zhirob AI, Kuznecov VYu, Subetto DA, Tide I, editors. *Geomorfologiya i Paleogeografiya Polyarnykh Regionov: Materialy sovmestnoi mezhdunarodnoi konferencii "Geomorphologiya i Paleogeografiya Polyarnykh Regionov", simposiuma "Leopoldina" i soveshchaniya rabochei gruppy INQUA Peribaltic*. Sankt-Peterburg, SPbGU, 9-17 sentyabrya 2012 goda. St Petersburg. p 467–68.

Mazurkevich AN, Dolbunova EV. 2012. The most ancient pottery and Neolithisation of Eastern Europe. *Fontes Archaeologici Posnanienses* 48: 143–59.

Meadows J, Bērziņš V, Lübke H, Schmölcke U, Zagorska I, Zariņa G. 2016. Dietary freshwater reservoir effects and the radiocarbon ages of prehistoric human bones from Zvejnieki, Latvia.

Journal of Archaeological Science: Reports. doi:10.1016/j.jasrep.2015.10.024.

Nedomolkina NG. 2004. Neoliticheskie kompleksy poselenii Veksa, Veksa III basseina Verkhnei Sukhony i ikh khronologiya [Neolithic complexes of the settlements Veksa, Veksa III of the Upper Sukhona basin and their chronology]. In: *Problemy khronologiya i etnokulturnykh vsaimodeistvii v neolite Evrazii* [Problems of chronology and ethno-cultural interrelations in the Neolithic of Eurasia]. Volume 2. St Petersburg. p 265–79.

Oshibkina SV. 2006. To the question of the Neolithic revolution and the Neolithization of the forest zone of Eurasia. In: Koryakova LN, Pavlov PY, Shirokov VN, Shorin AA, editors. *II Northern Archaeological Congress. Papers*. September 24–30, 2006, Khanty-Mansiisk. Ekaterinburg: Charoid. p 262–79.

Philippsen B. 2013a. The freshwater reservoir effect in radiocarbon dating. *Heritage Science* 1:24.

Philippsen B. 2013b. Der Süßwasser-Reservoireffekt in der 14C-Datierung: neue Analysen und mesolithische Kochexperimente. *Experimentelle Archäologie in Europa – Bilanz*. p 20–32.

Philippsen B, Meadows J. 2014. Inland Ertebølle Culture: the importance of aquatic resources and the freshwater reservoir effect in radiocarbon dates from pottery foodcrusts. In: Fernandes R, Meadows J, editors. Human Exploitation of Aquatic Landscapes' special issue. *Internet Archaeology* 37. http://dx.doi.org/10.11141/ia.37.91.

Philippsen B, Kjeldsen H, Hartz S, Paulsen H, Clausen I, Heinemeier J. 2010. The hardwater effect in AMS ^{14}C dating of foodcrusts on pottery. *Nuclear Instruments and Methods in Physics Research B* 268(7–8):995–8.

Piezonka H. 2008. Neue AMS-Daten zur frühneolithischen Keramikentwicklung in der nordosteuropäischen Waldzone. *Estonian Journal of Archaeology* 12(2):67–113.

Piezonka H. 2015. *Jäger, Fischer, Töpfer. Wildbeutergruppen mit früher Keramik in Nordosteuropa im 6. und 5. Jahrtausend v. Chr*. Archäologie in Eurasien 30. Bonn: Habelt-Verlag.

Piezonka H, Kostyleva E, Zhilin MG, Dobrovolskaya M, Terberger T. 2013. Flesh or fish? First results of archaeometric research of prehistoric burials from Sakhtysh IIa, Upper Volga region, Russia. *Documenta Praehistorica* XL:57–73.

Piličiauskas G, Heron C. 2015. Aquatic radiocarbon reservoir offsets in the southeastern Baltic. *Radiocarbon* 57(4):539–56.

Povlsen K. 2013. The introduction of ceramics in the Ertebølle Culture. *Danish Journal of Archaeology* 2(2):146–63.

Reimer PJ, Bard E, Bayliss A, Beck JW, Blackwell PG, Bronk Ramsey C, Grootes PM, Guilderson TP, Haflidason H, Hajdas I, Hatté C, Heaton TJ, Hoffmann DL, Hogg AG, Hughen KA, Kaiser KF, Kromer B, Manning SW, Niu M, Reimer RW, Richards DA, Scott EM, Southon JR, Staff RA, Turney CSM, van der Plicht J. 2013. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. *Radiocarbon* 55(4):1869–87.

Scharl S. 2004. *Die Neolithisierung Europas. Ausgewählte Modelle und Hypothesen*. Rahden: Verlag Marie Leidorf GmbH.

Schmölcke U, Meadows J, Ritchie K, Bērziņš V, Lübke H, Zagorska I. 2016. Neolithic fish remains from the freshwater shell midden Riņņukalns in northern Latvia. *Environmental Archaeology: The Journal of Human Palaeoecology*. doi:10.1179/1749631415Y.0000000011.

Sommer RS, Benecke N, Lõugas L, Nelle O, Schmölcke U. 2011. Holocene survival of the wild horse in Europe: a matter of open landscape? *Journal of Quaternary Science* 26(8):805–12.

Timofeev VI, Zaiceva GI, Dolukhanov PM, Shukurov AM. 2004. *Radiouglerodnaya khronologiya neolita Severnoi Evrazii* [Radiocarbon chronology of the Neolithic of Northern Eurasia]. St Petersburg.

Utkin AV, Kostyleva EL. 2001. Pogrebeniya na stoyanke Karavaikha [Burials at the site of Karavaikha]. *Rossiiskaya arkheologiya* 3:55–66.

Vybornov A, Zaitseva G, Kovaliukh N, Kulkova M, Possnert G, Skripkin V. 2012. Chronological problems with neolithization of the northern Caspian Sea area and the forest-steppe Povolzhye region. *Radiocarbon* 54(3–4):795–9.

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.

Yanits LY. 1976. Raskopki neoliticheskogo poseleniya Kyaepa (Excavation of the Neolithic settlement of Kääpa). Eesti Teaduste Akadeemia Toimetised 25. Ühistkomateadused. p 45–8.

Yoshida K, Kunikita D, Miyazaki Y, Nishida Y, Miyao T, Matsuzaki H. 2013. Dating and stable isotope analysis of charred residues on the Incipient Jomon pottery (Japan). *Radiocarbon* 55(2–3):1322–33.

Zaitseva G, Skripkin V, Kovaliukh N, Possnert G, Dolukhanov P, Vybornov A. 2009. Radiocarbon dating of Neolithic pottery. *Radiocarbon* 51(2):795–801.