Radiocarbon, Vol 57, Nr 4, 2015, p 611–623 DOI: 10.2458/azu_rc.57.18427

LATE NEOLITHIC SUBSISTENCE STRATEGY AND RESERVOIR EFFECTS IN ^{14}C DATING OF ARTIFACTS AT THE PILE-DWELLING SITE SERTEYA II (NW RUSSIA)

M Kulkova[1,2] • A Mazurkevich[3] • E Dolbunova[3] • M Regert[4] • A Mazuy[4] • E Nesterov[1] • M Sinai[1]

ABSTRACT. Radiocarbon dating and research into offset correction for freshwater reservoir effect were conducted at the pile-dwelling site Serteya II, located in the Dvina-Lovat' basin (northwestern Russia). Cultural layers of this site are situated underwater, hence the unique state of preservation of material culture of the 3rd millennium cal BC. ^{14}C dating of different organic materials [wood, hazelnut (*Corylus avellana*), and elk bones] from this site allows their ages to be correlated and ^{14}C age offsets caused by freshwater reservoir effects (hardwater effects) in the dating of materials such as organic crust, pottery, bones, and lake sediments to be estimated. Consideration of the late Neolithic subsistence strategy underpinning the archaeological finds from this site and analysis of lipid components in ceramic vessels, as well as the determination of ^{14}C activity of modern aquatic and terrestrial samples, allows us to calculate the local freshwater reservoir effect and ^{14}C age offset for charred food crusts from different ceramic vessels more precisely.

INTRODUCTION

The problem of radiocarbon dating of organic matter that could include organic components of aquatic origin has been discussed widely in recent years. It is especially important for the dating of materials such as human and animal bones, whose diet could have included marine and freshwater fish, mollusks, or aquatic plants, as well as organic crusts on pottery in which these types of food could have been prepared.

Along with the well-known marine reservoir effect, freshwater reservoir effects (FREs) such as the hardwater effect, have attracted a great deal of attention. Corrections for FREs can be calculated based on additional measurements of ^{14}C activity in modern aquatic plants, sediments, and living organisms, e.g. fish. However, the scale of reservoir effects may vary within the same water body, as shown by Philippsen (2013). FREs may vary from 0 to >2000 yr and even more for different water basins (Deevey et al. 1954; Broecker and Walton 1959; Andree et al. 1986; Cook et al. 2001; Fischer and Heinemeier 2003; Grimm et al. 2009; Smits and van der Plicht 2009).

FRE correction is especially important for dating of organic crust on pottery walls, in cases where freshwater products were part of the diet. Different products could have been cooked in the same vessel, however—not only fish and aquatic food, but also plants and animals of terrestrial origin. As shown by Hart (2014), the proportion of each product included in the organic crust should be taken into account, as the contribution of freshwater products to the organic crust can be much less than 100%. In such cases, the appropriate FRE correction would be much lower than the local FRE. In order to solve this task, Hart proposed several models of calculation of FRE correction for organic crusts, considering the mean composition of fats, proteins, and carbohydrates in food. One of the problems of such an approach is the difficulty of estimating the percentage of every product included as a component in organic crust, which might lead to inaccurate calculation of FRE correction.

^{14}C dating and research into local FREs was conducted for archaeological sites in the Dvina-Lovat' basin, on the border of the Pskov and Smolensk regions in northwestern Russia (Figure 1). Materials from the Serteya II site, with remains of pile dwellings dated to the end of the Middle–Late Neolithic (3rd millennium cal BC), became the main subject for this research.

1. Herzen State Pedagogical University, Saint Petersburg, Russia.
2. Corresponding author. Email: kulkova@mail.ru.
3. The State Hermitage Museum, Saint Petersburg, Russia.
4. UMR 7264 UNS – CNRS, CEPAM, Nice, France.

Proceedings of *Radiocarbon and Diet: Aquatic Food Resources and Reservoir Effects*
First International Conference, 24–26 September 2014, Kiel, Germany
Edited by Ricardo Fernandes, John Meadows, and Alexander Dreves

Because the cultural layers of the Serteya II site are underwater, the material culture is found in a unique state of preservation. Several wooden pile dwellings were found here (Figure 2), and a detailed chronological scheme of their construction, based on ^{14}C dating, was elaborated (Zaitseva et al. 2003). ^{14}C dating of different organic materials [wood, hazelnut (*Corylus avellana*), animal bones] from this site allows estimation of the offset caused by FREs in the dating of materials such as organic crust, pottery, fish bones, and lake sediments. These materials are considered to be contemporaneous since they come from a single cultural layer formed here over a short period of time, before being covered by later sediments (see Table 1: SPb-1404, a ^{14}C date made on the overlying silt).

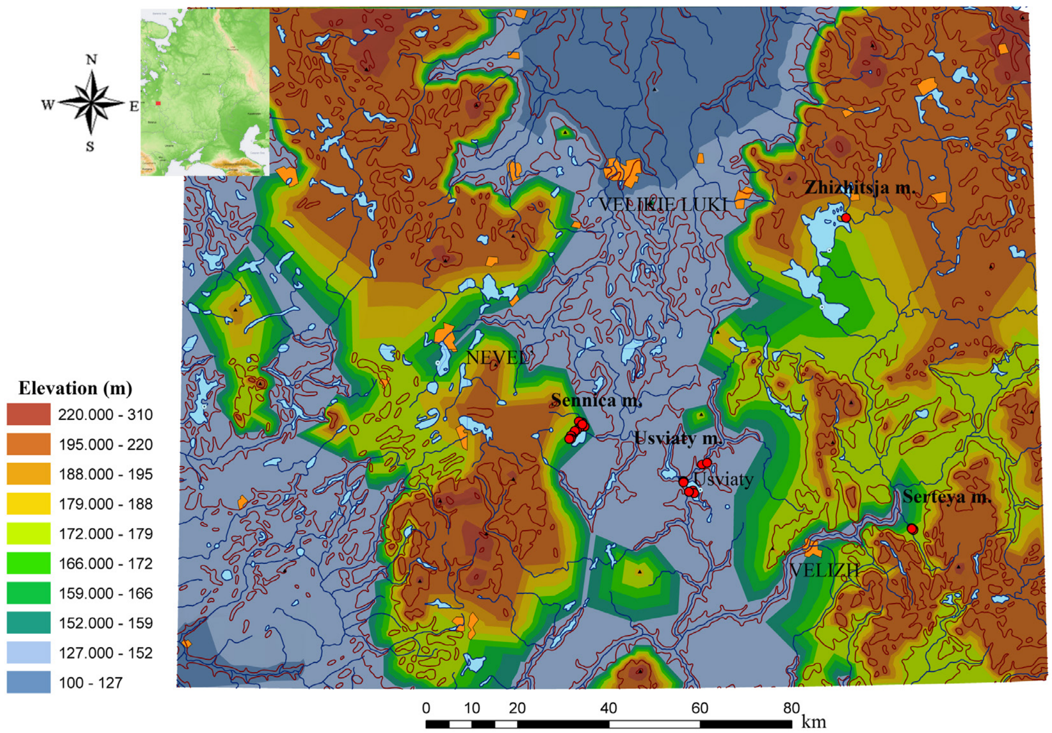

Figure 1 Location of Dnepr-Dvina region and the Serteysky microregion

STUDY AREA AND CHRONOLOGICAL BACKGROUND

Serteya II is located on the shore of the Serteyka River, a small tributary of the Western Dvina River (Smolensk region), in the Dnepr-Dvina region. The chain of lakes in this area was formed at the end of the Pleistocene to the beginning of the Holocene periods, within parts of fluvioglacial and morainic-hill relief, after the retreat of the Late Würm stage ice sheet. Currently, the lake basins are waterlogged and are cut by the narrow course of the Serteyka River. Two main lake basins can be distinguished: the large Serteyskaya Lake basin and the smaller Nivnikovskaya Lake basin. The Serteyskaya lake basin was formed by reddish-red morainic clay loams. The Nivnikovskaya Lake basin was formed by fluvioglacial kame sediments including fine-stratified well-sorted yellow and yellow-pink sands. Morainic clay loams contained carbonates, which could influence the isotopic composition of dissolved inorganic carbonate (DIC) in the Serteyskaya Lake basin. The CaO content of sandy lake sediments at the site Serteya X, located on the shore of the Serteyskaya basin, not far from Serteya II site, ranges from 6.8 to 0.5%, compared to 30 to 3.8% in gyttja sediments. The gyttja

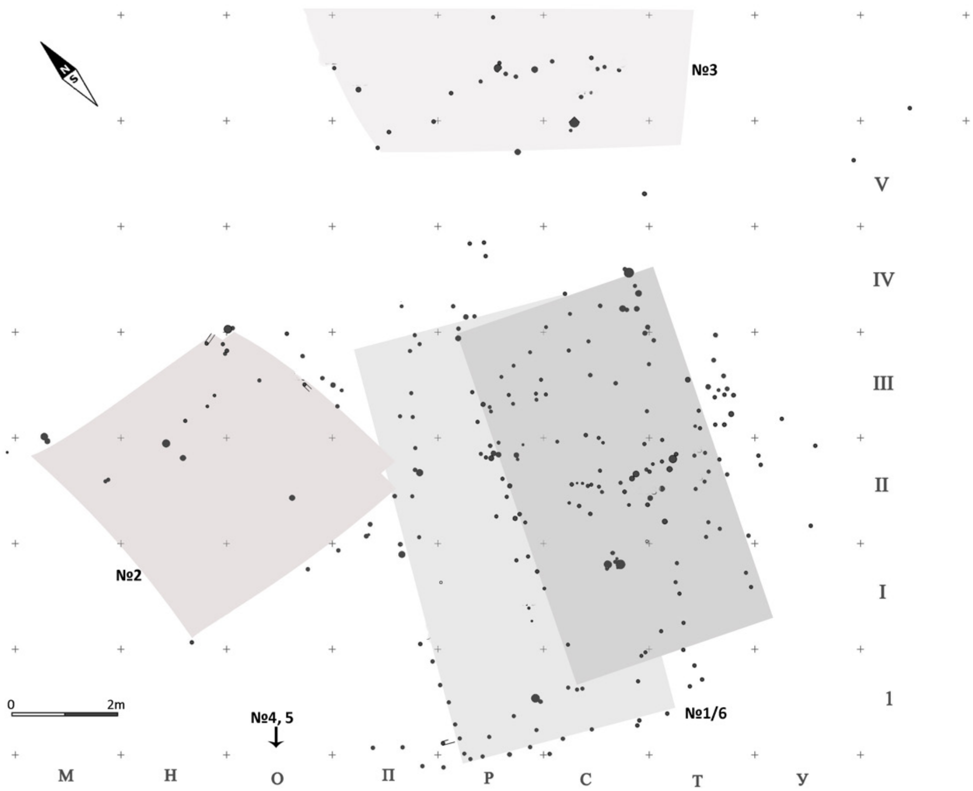

Figure 2 Distribution of pile dwellings on the site Serteya II with wooden piles' indication

layer with mollusk shells is most enriched in carbonates (Kulkova and Savelieva 2003), suggesting that the calcite was precipitated from the lake water where the carbonate was once in solution.

^{14}C dating of wooden piles of different constructions was carried out in order to develop an absolute chronology for Serteya II. A total of 49 dates was obtained (Zaitseva et al. 2014). These dates were statistically treated, and a mean age was determined for the site and for the dwellings (Davison et al. 2009; Mazurkevich et al. 2009). The most ancient construction, Nr 4, attributed to the late stage of the Usviatskaya culture, appeared in the first half of the 3rd millennium cal BC, based on ^{14}C dates made on the wooden remains of the dwellings (Mazurkevich et al. 2010; Zaitseva et al. 2014). The site was then probably uninhabited for some time. The next period of building activity is dated to 2570–2330 cal BC (2σ), when dwelling Nr 2 was erected. The active period of settlement can be traced to 2470–2270 cal BC (2σ), when dwellings Nr 1 (Nr 6) and Nr 3 were constructed. A small community is thought to have lived here during this period of time, and to have successively erected and/or reconstructed pile dwellings on the same places. Also, there are several dates made on wooden piles, attributed to 2210–2020 cal BC (2σ) and 1920–1730 cal BC (2σ), which imply that the site was also inhabited and used later. ^{14}C dates of piles from the central part of the site form several groups of dates within the interval 2800–1700 cal BC (2σ). The ^{14}C dates correlate with stratigraphic observations, as four construction horizons can be distinguished here. The results of dendrochronological analysis show that these structures were reconstructed/enlarged every 5 and 17 yr (Hookk 2014). Thus, the ^{14}C dates on organic crust for different types of pottery, coming from different horizons, can be correlated with periods of inhabitation at the site.

https://doi.org/10.2458/azu_rc.57.18427 Published online by Cambridge University Press

Differences distinguished in pottery technology, decor, and morphology can be explained by the chronological difference between assemblages. Pottery attributed to the middle Neolithic Usviatskaya culture (second half of the 4th to first half of the 3rd millennium cal BC) and Funnel Beaker culture can be found in the earliest dwellings Nr 4 and Nr 2, and in the earliest horizon of dwelling Nr 1. The later influence of the Dnepr-Donetsk culture (mid-4th to second half of the 3rd millennium cal BC; Jaziehienko and Jozwiak 2004) can be traced. The influence of Corded Ware culture can also be seen in assemblages from dwellings Nr 1 and Nr 3 (Mazurkevich et al. 2010).

METHODS

Our analysis of ^{14}C dates took into account the determination of FRE based on ^{14}C dating modern materials, statistical treatment of ^{14}C dates, and lipid analysis of organic crusts. At the same time, we considered archaeological analysis and treatment of materials based on stratigraphic observation and typological-technological research of materials, culminating in the development of a relative chronology of different ceramic types.

One of the most important assumptions is that the construction date of the dwelling is contemporaneous with pottery and other materials found inside the dwellings. Those artifacts dating to preceding periods might indicate earlier periods of activity on these parts of the site or the earlier dates may be due to offsets caused by FRE, which can be determined for pottery based on the analysis of its morphological, technological, and décor characteristics, which changed through time, as well as their stratigraphic position in the whole complex.

Samples of organic crust on pottery found in different pile dwellings were used for ^{14}C dating (Figure 3). Samples of modern terrestrial plants, aquatic samples of cow lily (*Nuphar lutea*) roots, stems and leaves, and fish (*Squalius cephalus*) were collected at the site of the Serteya II underwater excavations in order to estimate the hardwater effect. ^{14}C dating by the traditional conventional method was undertaken by measurement using a Quantulus 1220 liquid scintillation counter (Table 1). Samples were pretreated using standard methods (Nakamura et al. 2001; Boudin et al. 2010). Also, measurements of carbon stable isotopes were made for organic crust samples (Table 1) using the Nu Horizon IRMS instrument.

Samples of organic crust attributed to ceramic types of the Zhizhitskaya culture were chosen for lipid analyses, the results of which might characterize vessels' use during this period. In order to assess the degree of preservation of lipid components in ceramic vessels, a series of 18 carbonized residues on ceramic sherds was investigated chemically (Table 2) following a procedure now currently used on amorphous organic residues from pottery (see e.g. Charters et al. 1995). Briefly, an amount between 42 and 116 mg was ground with a mortar and pestle, and ultrasonically extracted (2 × 15 min) with 10 mL of a mixture of chloroform:methanol (2:1 v/v). An internal standard (n-C_{34}) was added to the solution at a concentration of 1 mg/mL. After centrifugation, the supernatant was concentrated under a gentle stream of nitrogen at 40°C; an aliquot (100 μL versus 500 μL) was then trimethylsilylated and analyzed by gas chromatography (HP6890 GC equipped with an on-column injector and a Chrompack Sil 5 CB LB/MS column: 15 m × 0.32 mm i.d.; 0.1-μm-thick film). The oven temperature was ramped from 50°C (held isothermal for 2 min) to 350°C in 10°C/min steps (and held isothermal for 10 min). The analysis was performed using helium as carrier gas, with the following programmed flow: 2 mL min^{-1} for 17 min, then 4 mL min^{-1} (ramp of 1 mL min^{-2}) for 5 min, and 6 mL min^{-2} (ramp of 1 mL min^{-2}) for 15 min. Gas chromatography-mass spectrometry (GC-MS) analyses performed on three samples (MR1913, MR1919, and MR1921) were carried out in the same conditions as described for GC analysis (except the use of a split/splitless injection system operating in the splitless mode), with a Shimadzu QP2010 ultra. The GC temperature program was 1 min at 50°C, 15°C min^{-1} until 100°C, 10°C min^{-1} until 240°C, 20°C min^{-1} until 380°C, and 7 min at 380°C. Mass spectra were acquired using electron ionization at 70 eV. The mass range was scanned from *m/z* 50–950 in 0.6 s. The temperature of the ion source was fixed at 200°C and for the transfer line at 300°C.

Table 1 Radiocarbon dates made on modern and ancient organogenic materials from the site Serteya II.

Lab code	Material	pMC	Conventional ^{14}C age (BP)	Reservoir correction (^{14}C yr)	δ^{13}C (‰ VPDB)
SPb-1398	Grass	102.5 ± 0.60	Modern age		–26.23 ± 0.05
SPb-1399	Root of cow lily	98.8 ± 0.60	96 ± 60	295 ± 68	–25.45 ± 0.05
SPb-1400	Cow lily stem	99.9 ± 0.60	7 ± 60	206 ± 67	–25.85 ± 0.05
SPb-1401	Cow lily leaf	103.9 ± 0.60	Modern age	–109 ± 66	–25.25 ± 0.05
SPb-1402	Modern fish	95.3 ± 0.60	386 ± 60	585 ± 69	n/d
SPb-1403	Hazelnut shells	—	3826 ± 100		n/d
SPb-1404	Silt above cultural layer	—	2452 ± 100		n/d
SPb-1181 ***(MR 1911)***	Organic crust on the vessel (239-3/11/17, 239-3/11/30), Zhizhitskaya culture, construction 1/2	—	4080 ± 120	189 ± 121	–30.5 ± 0.07
SPb-1184	Organic crust on the vessel of Zhizhitskaya culture, construction 1, constructive horizon 2	—	3970 ± 120	79 ± 121	–27.8 ± 0.07
SPb-1193	Organic crust on the vessel fragment, Zhizhitskaya culture, construction 1, constructive horizon 3	—	3992 ± 120	101 ± 121	–29.83 ± 0.07
SPb-1182	Organic crust on the vessel, late stage of Usviatskaya culture, construction 1	—	4260 ± 120	Probable value in the interval 0–585	–31.5 ± 0.07
SPb-1183	Organic crust on the vessel, Zhizhitskaya culture, construction 2	—	3880 ± 120		–31.7 ± 0.07
SPb-1179	Organic crust on the vessel, Zhizhitskaya culture, construction 3	—	4200 ± 120		–31.1 ± 0.07
SPb-1180 ***(MR 1921)***	Organic crust on the vessel (310- 2/67 (1,2), 306-3/3(1)), Zhizhitskaya culture, construction 1, constructive horizon 1/2	—	3977 ± 120	86 ± 121	–30.5 ± 0.07
SPb-1195	Collagen from fish bones, sq. C-t/ii, construction 1, horizon 3	—	3929 ± 120	38 ± 121	–26.13 ± 0.05
SPb-1191	Organic crust on the vessel, late stage of Usviatskaya culture, construction 1, constructive horizon 3	—	4642 ± 150	Probable value in the interval 0–585	–31.6 ± 0.07
SPb-1192	Organic crust on the vessel, late stage of Usviatskaya culture, construction 1, constructive horizon 3	—	4020 ± 150	Probable value in the interval 0–585	–29.9 ± 0.07
SPb-1204	Fish bones, sq. O/iii, construction nr 2	—	4611 ± 120		
Le-10413	Elk bone, sq. T/iii, construction nr 1, horizon 3	—	3920 ± 100		

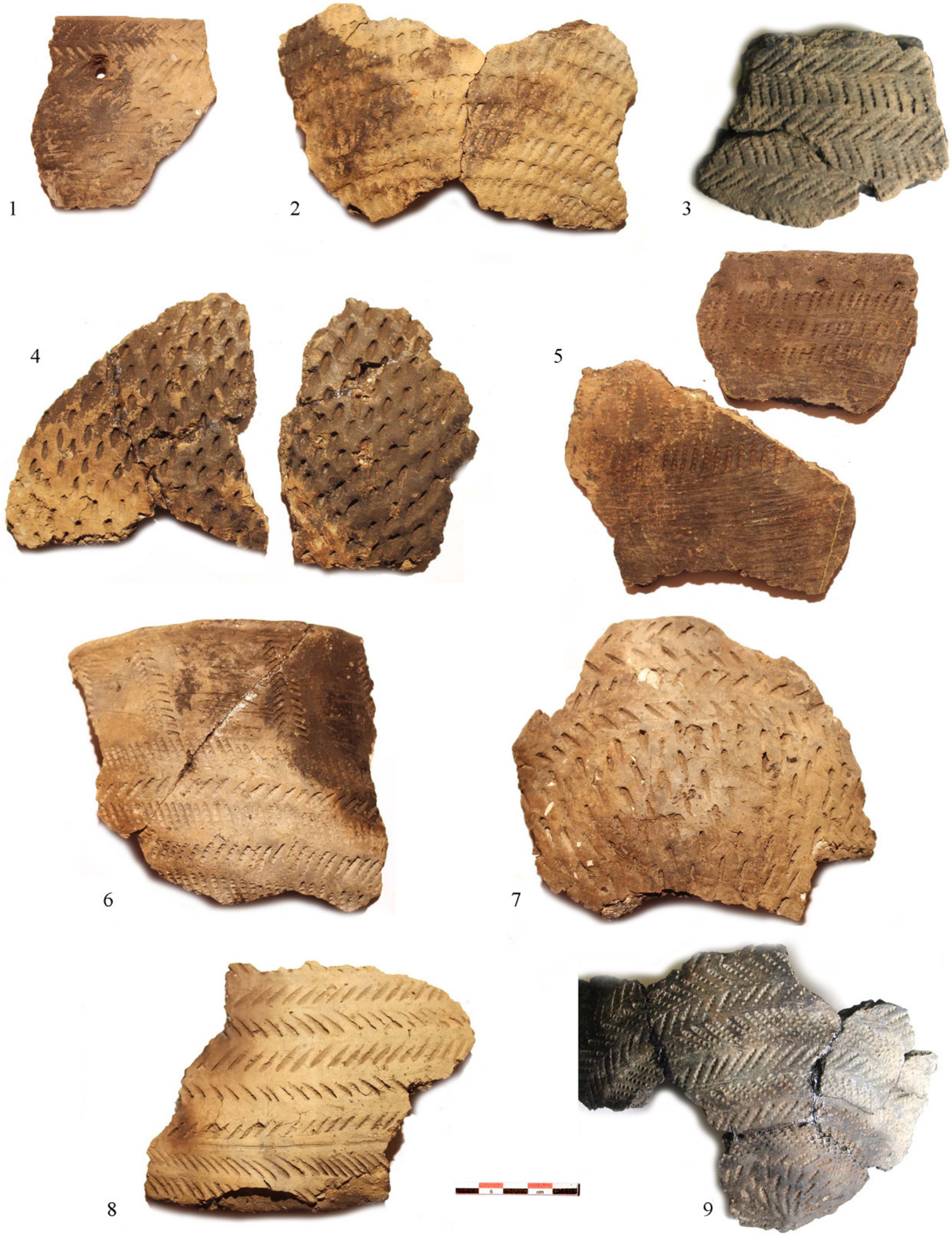

Figure 3 Pottery fragments from the Serteya II site. The lab indexes of ^{14}C dates are as follows: 1 – SPb-1191; 2 – SPb-1181; 3 – SPb-1179; 4 –SPb-1183; 5 – SPb-1192; 6 – SPb-1182; 7 – SPb-1193; 8 – SPb-1184; 9 – SPb-1180.

Lipid constituents were detected in all samples (Figure 4a,b), revealing in many cases an excellent preservation. The concentration of the total lipid extract (TLE) was between 76 μg/g and more than 2 mg/g, which is very high compared to that found in previous studies (Evershed 2008, 2009; Regert 2011). Furthermore, in six samples, triacylglycerols (TAG) were preserved, representing up to 30% of

https://doi.org/10.2458/azu_rc.57.18427 Published online by Cambridge University Press

the TLE (Figure 5). These data demonstrate excellent conservation of the lipid extract, as already observed in other anaerobic contexts (Regert et al. 2001; Regert and Mirabaud 2014).

Table 2 List of samples analyzed for their total lipid extract.

Sample nr	Lab nr	Part of the vessel	C of TLE	C_{16}/ C_{18}	% TAG	Preliminary interpretation
11	MR1910	near bottom	427	1.8	0	Degraded animal fats (saturated fatty acids C_{16} and C_{18} and cholesterol + MAG)
12	MR1911	wall	76	3.4	0	Mixture of plant and animal fats? (cholesterol, stigmasterol and $C_{16/18}$ = 3,4 with presence of unsaturated C_{18} fatty acids)
13	MR1912	wall	272	3.3	0	Mixture of vegetable and animal fats? (cholesterol, stigmasterol and $C_{16/18}$ = 3,3 with presence of unsaturated C_{18} fatty acids) + presence of even numbered long-chain fatty acids C_{22}–C_{26} and of $C_{24:1}$, which may indicate the exploitation of aquatic resources
14	MR1913	wall	192	1.9	?	Fatty substance with unidentified long-chain compounds, presence of cholesterol and stigmasterol and of $C_{18:1}$, which may indicate a mixture of animal and plant origin
15	MR1914	wall	140	2.2	0	Unidentified fatty substance
16	MR1915	wall	187	2.7	?	Unidentified fatty substance
17	MR1916	wall	240	2	<5	Degraded animal fat
18	MR1917	near bottom	1508	0.9	25	Degraded animal fat with 25% of TAG (C_{46}–C_{54}) => subcutaneous animal fats.
19	MR1918	wall	83	3.6	0	Unidentified fatty substance
20	MR1919	wall	650		0	Mixture of fatty acids, diterpenoids, triterpenoids, and long-chain unidentified compounds => plant residues
21	MR1920	wall	384	1.6	<5	Degraded animal fat
22	MR1921	wall	606	0.6	41	Subcutaneous animal fats (fatty acids, MAG, DAG and 41% of TAG C_{46}–C_{54}) with presence of plant biomarkers (triterpenoids under interpretation)
23	MR1922	near bottom	443	1.7	11	Degraded subcutaneous animal fats (11% TAG from C_{46} to C_{52})
24	MR1923	rim	497	1.9	<5	Unidentified fatty substance
25	MR1924	wall	2545	1.5	8	Subcutaneous animal fats (fatty acids, cholesterol and 8% of TAG C_{46}–C_{54})
26	MR1925	wall	166	2.1	0	Unidentified fatty substance
27	MR1926	wall	210	1.3	30	Subcutaneous animal fats (fatty acids, cholesterol and 30% of TAG C_{46}–C_{54})
28	MR1927	wall	933	2.4	8	Subcutaneous animal fats (fatty acids, cholesterol and 8% of TAG C_{46}–C_{54})

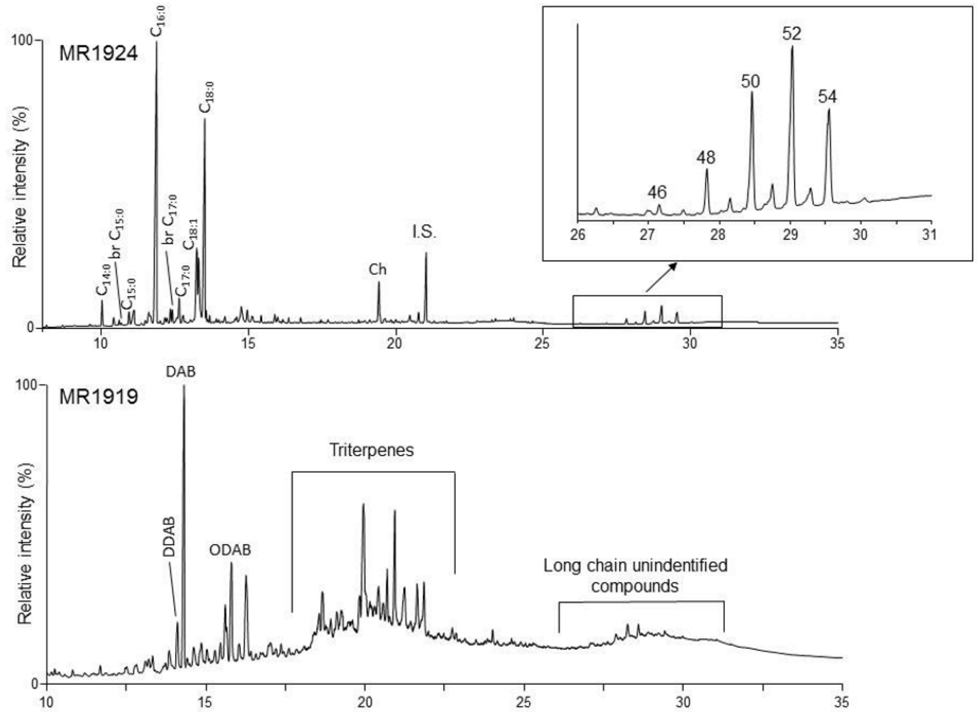

Figure 4 (a) Chromatogram of sample MR1924 showing a profile typical of animal fat; (b) chromatogram with a mixture of diterpenoids of sample MR1919 (DDAB = dehydro-dehydroabietic acid; DAB = dehydroabietic acid; and ODAB = 7-oxodehydroabietic acid) and triterpenoids.

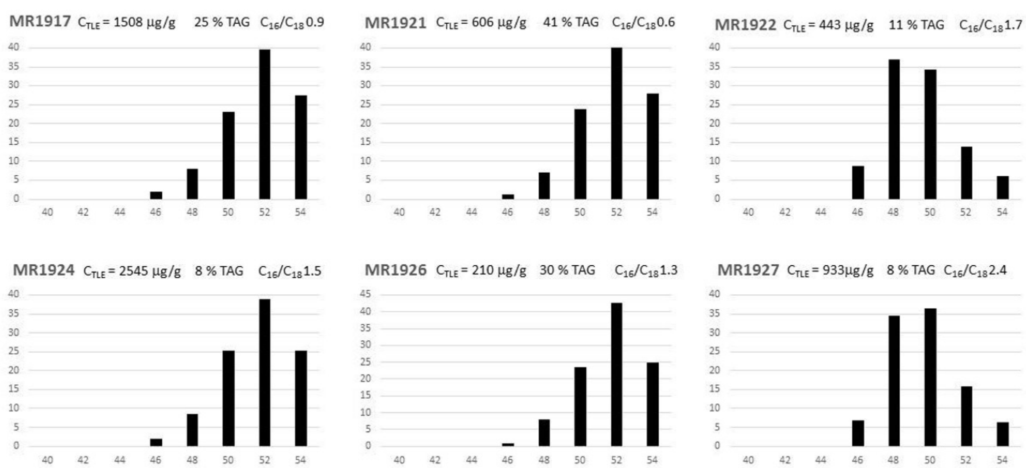

Figure 5 TAG distribution in six samples from Serteya II

RESULTS

Subsistence Strategy and Ancient Diet

Research on the diet and subsistence strategy of ancient inhabitants on this territory plays an important role in the analysis of ^{14}C dates and an interpretation of ^{14}C dating results. Spatial analysis

https://doi.org/10.2458/azu_rc.57.18427 Published online by Cambridge University Press

of lacustrine pile dwellings in the study area reveals a clear subsistence pattern based on catchment area, which limits foraging to a 2-hr walking distance (~10 km) from the central hunting lodge (Zvelebil 1996). The catchment area of the pile dwellings includes several distinct landscape types, a combination that made possible a productive hunter-gatherer economy and strongly contributed to the settlement system at this time (Dolukhanov and Miklyaev 1986).

The faunal and botanical records indicate that the lacustrine sites' dwellers relied heavily on wild resources, with year-round procurement of meat and fur animals as well as fishing. The majority of bones found at the pile dwellings belonged to elk, with fewer examples coming from other species (Sablin and Siromyatnikova 2009). Birds were also hunted. The number of fish bones and fragments of *Unio* shells recovered indicates the considerable economic significance of aquatic resources. Also, coprolites of wild boar, filled with fish scales and bones, were found on the site Serteya II, which could indicate that they were held for some time on the site. That is why FRE correction should be taken into account when dating bones of these wild pigs. Food gathering was of considerable significance as well; a considerable quantity of shells from hazelnuts, water chestnut, and acorns were recovered. At least 30 edible wild plants were used for food. Processed hazelnut and water chestnut may have been a surrogate for bread and the main source of plant protein.

Traces of agriculture can be also observed in the early Subboreal (Sb-1, 3350–2600 cal BC) period, due to *Cerealia* identification by pollen analysis and finds of ploughs (Mazurkevich and Dolbunova 2011). Analysis of the paleolandscape suggests that at the beginning of the Subboreal, fertile soils along the margins of the lakes were used as fields for crops and cattle herding. The chemical composition of gyttja and coastal sandy clay sediments also suggest the appearance of agricultural activity.

Subsistence strategies might give evidence for variability in the ancient diet. The variety in the ceramic assemblage on the site Serteya II, with vessels of various forms and volumes, with or without traces of organic crust, might indicate different pottery functions: the existence of kitchen ware for cooking different meals, in notably different ways (which might be evidenced by the existence of pointed vessels with a hole in the bottom, conical and flattened bottoms of vessels), storage ware of big vessels 30–40L in volume, and tableware including dishes, small bowls, and small cups.

Although work on lipid constituents from charred organic crusts is still in progress, the preliminary results show an interesting diversity of natural products processed in the vessels. First of all, the TAG in all the samples have a narrow distribution, peaking at C_{52} in most cases, compatible with subcutaneous animal fats, for which the origin has still to be determined, considering the importance of hunting at the site of Serteya II (Figure 5).

In one case (sample MR1921), the presence of plant biomarkers from the triterpenoid family indicates a mixture with plant substances. In three samples (MR1911, MR1912, and MR1913), the presence of fatty acids with cholesterol and stigmasterol may indicate a mixture of plant and animal fats. The presence of saturated long-chain even-numbered fatty acids (C_{22}–C_{26}), together with the presence of $C_{24:1}$, could be related to the consumption of aquatic resources. For the other samples, animal fats are probably the main commodities processed in the vessels, except for sample MR1919 (Figure 4b). Indeed, the presence of complex mixtures containing diterpenoids of the Pinaceae family together with triterpenoid biomarkers may clearly be related to plant materials. It thus appears that organic matter of lipid origin is very well preserved in pottery from Serteya II, and it suggests evidence for the exploitation of a large range of natural resources of various origins, for which further analyses are now necessary to determine their exact nature (GC-IRMS data and complete molecular investigation by GC-MS).

Analysis of carbon stable isotopes showed bulk organic crusts have depleted $\delta^{13}C$ values, from –27.8 to –31.5‰, which might reflect the use of freshwater products in the vessels, although these results do not exclude traces of terrestrial animals and vegetation. This corresponds to the analysis of lipid content in organic crust by gas chromatography, which showed that different types of food were prepared in the vessels, with terrestrial animal, plant, and aquatic origin.

Analysis of Radiocarbon Dates

The present hardwater effect for the Serteyka River was calculated according to the formula proposed by Philippsen (2013): $R = 8033 \times \ln(pMC_T/pMC_A)$, where pMC_A = percentage of modern ^{14}C content in water samples and pMC_T = percentage of modern ^{14}C content in terrestrial samples. The ^{14}C activity for modern samples is presented in the Table 1. The reservoir effect in the fish sample was calculated as 585 ^{14}C yr, while different parts of cow lily had reservoir effects from 206 to 295 ^{14}C yr.

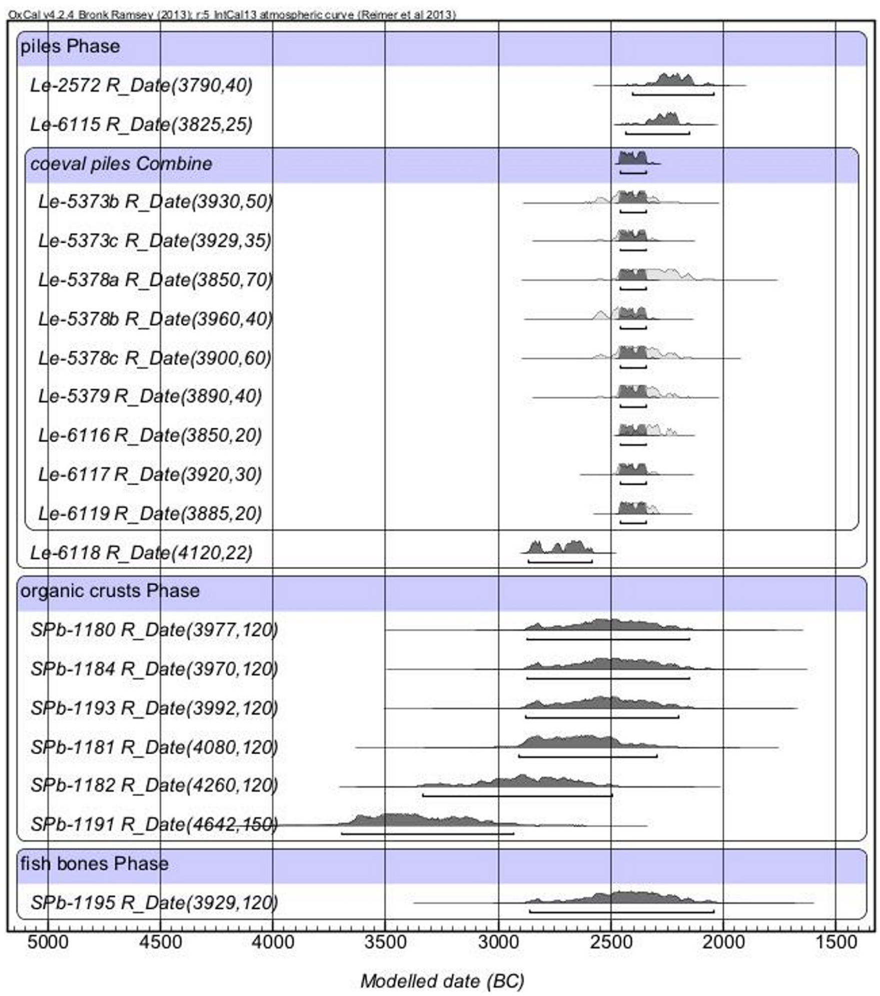

Figure 6 Calibration and analysis by Markov chain Monte Carlo (MCMC) method of ^{14}C data from construction 1 for piles (^{14}C results from Zaitseva et al. 2014) and organic crust and fish bones (Table 1). The pile results included in the OxCal Combine function were consistent with a single date, which we regard as the best estimate for the construction of this pile dwelling.

^{14}C dates made on wood and organic crust were treated by the Markov chain Monte Carlo (MCMC) method in order to summarize the dates that can be best attributed to the construction 1 (Figure 6). Also, the results of typological-technological analysis and stratigraphic observation of material distribution were taken into account, which is why the FRE correction was not "automatically" included in this model, as the organic crust dates do not contradict the dates of timbers in the synchronous pile dwelling. Therefore, any FRE must have been rather low.

The calibrated age for a statistically homogeneous selection of dates for construction periods 1–3 of dwelling Nr 1 lies in the interval 2470–2300 cal BC (2σ) (Figure 7). Thus, dates made on two organic crusts, 4642 ± 150 BP (SPb-1191) and 4260 ± 120 BP (SPb-1182), on vessels found in dwelling Nr 1 appear to be much older. However, taking into account that this type of pottery is attributed to the late stage of the Usviatskaya culture, just before the Zhizhitskaya culture, the FRE incorporated in these dates might be not very high, as most dates for dwelling Nr 1 can be attributed to the Zhizhitskaya culture. FRE estimates for the dates on organic crust from the Zhizhitskaya culture sherds in construction 1 (calculated by comparing their ^{14}C ages to the weighted mean of the timber dates, 3891 ± 11 BP) are not statistically significant (Table 1). FRE estimates for the dates made on organic crust from Usviatskaya culture vessels should be calculated in comparison to dates on parts of construction Nr 1 attributed to the late stage of Usviatskaya culture, which were uncovered only recently.

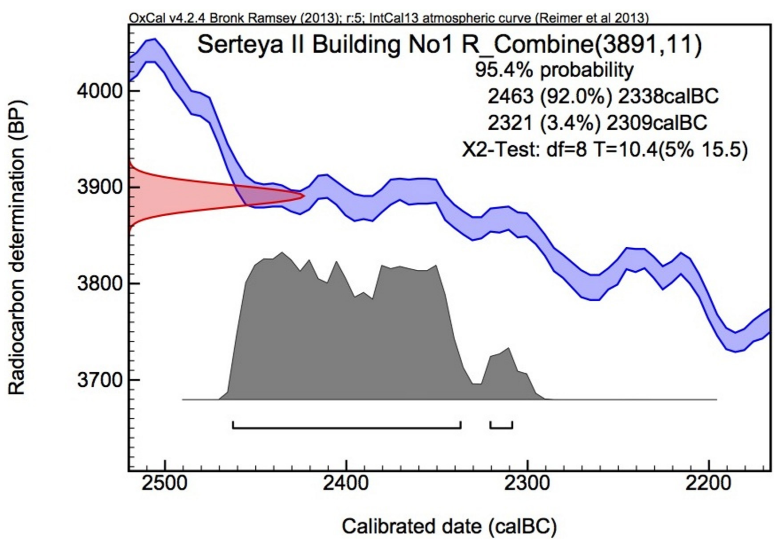

Figure 7 Calibration of the weighted mean of contemporaneous piles in pile-dwelling Nr 1 (see Figure 6 for individual measurements included).

CONCLUSION

In light of the presented results, we could calculate the FRE for organic crust samples from every vessel, found in every pile dwelling, by subtracting the combined ^{14}C ages for piles definitely related to construction from the ^{14}C age of the organic crust. These offsets are consistent with the FREs obtained for modern water vegetation and fish from this freshwater basin. Food composition, more particularly, the amount of carbon derived from fish in food crust, affects the interpretation of organic crust ^{14}C ages. The FRE offsets proposed for food crust samples are shown in the Table 1. In some cases, e.g. when the amount of carbon derived from aquatic ingredients is small, the FRE will be negligible as well.

https://doi.org/10.2458/azu_rc.57.18427 Published online by Cambridge University Press

In such an approach, in which different organic materials from one site with brief periods of occupation are dated, Bayesian statistical modeling may be used to interpret ^{14}C results made on tree rings and allow FREs incorporated in the ^{14}C ages of organic crusts to be identified and quantified, or disregarded, based also on archaeological evidence, and not only on comparison of dates made on different materials. The results may allow us to estimate the amount of freshwater food in the composition of food crust. Although the species of fish found in Neolithic layers are known, we do not know which types of fish were prepared in pottery vessels.

^{14}C dates on ancient organic materials from the site Serteya II (charred food crust on pottery, fish, and animal bones) and their comparison with the dates on wooden piles show in most cases negligible reservoir effects, which is interesting evidence considering the determination of FRE in modern materials from this region. This approach to identifying FREs in the Serteysky archaeological microregion could be also applied in the interpretation of ^{14}C dates on charred food crust from vessels dated to different chronological periods. Values obtained for reservoir effect error correction can be used only for this microregion. Further research to identify reservoir effects in this lake basin, as well as neighboring basins in different geological settings, must be conducted in order to refine ^{14}C dates of artifacts and chronological scheme of cultures' development with the dating of charred food crust and/or bone material.

ACKNOWLEDGMENTS

The researches were supported by RFBR, project 13-06-12057. We would like to thank Dr John Meadows for his valuable corrections proposed for this paper. Lipid analyses were funded by the Mission à l'Interdisciplinarité from the CNRS and we are grateful for their support in this project.

REFERENCES

Andree M, Oeschger H, Siegenthaler U, Riesen T, Moell M, Ammann B, Tobolski K. 1986. ^{14}C dating of plant macrofossils in lake sediment. *Radiocarbon* 28(2A):411–6.

Boudin M, Van Strydonck M, Crombé P, DeClercq W, Dierendonck RM, Jongepier H, Ervynck A, Lentacker A. 2010. Fish reservoir effect on charred food residue ^{14}C dates: Are stable isotope analyses the solution? *Radiocarbon* 52(2–3):697–705.

Broecker WS, Walton A. 1959. The geochemistry of C^{14} in the freshwater systems. *Geochimica et Cosmochimica Acta* 16(1):15–38.

Charters S, Evershed RP, Blinkhorn PW, Denham V. 1995. Evidence for the mixing of fats and waxes in archaeological ceramics. *Archaeometry* 37(1):113–27.

Cook GT, Bonsall C, Hedges REM, McSweeney K, Boroneant V, Pettitt PB. 2001. A freshwater diet-derived ^{14}C reservoir effect at the stone age sites in the Iron Gates Gorge. *Radiocarbon* 43(2A):453–60.

Davison K, Dolukhanov PM, Sarson GR, Shukurov A, Zaitseva GI. 2009. Multiple sources of the European Neolithic: mathematical modelling constrained by radiocarbon dates. In: Dolukhanov PM, Sarson GR, Shukurov AM, editors. *The East European Plain on the Eve of Agriculture*. *BAR International Series 1964*. Oxford: Archaeopress. p 197–211.

Deevey Jr ES, Gross MS, Hutchinson GE, Kraybill HL. 1954. The natural ^{14}C contents of materials from hard-water lakes. *Proceedings of the National Academy of Sciences of the USA* 40(5):285–8.

Dolukhanov PM, Miklyayev AM. 1986. Prehistoric lacustrine pile dwellings in the north-western part of the USSR. *Fennoscandia Archaeologica* 3:81–9.

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

Evershed RP. 2009. Compound-specific isotopes in organic residue analysis in archaeology. In: Colombini MP, Modugno F, editors. *Organic Mass Spectrometry in Art and Archaeology*. Chichester: Wiley. p 391–432.

Fischer A, Heinemeier J. 2003. Freshwater reservoir effect in ^{14}C dates of food residue on pottery. *Radiocarbon* 45(3):449–66.

Grimm EC, Maher Jr LJ, Nelson DM. 2009. The magnitude of error in conventional bulk-sediment radiocarbon dates from central North America. *Quaternary Research* 72(2):301–8.

Hart JP. 2014. A model for calculating freshwater reservoir offsets on AMS-dated charred, encrusted cooking residues formed from varying resources. *Radiocarbon* 56(3):981–9.

Hookk DY. 2014. Fuzzy logic application to the dendrochronological analysis of the constructions on the pile-dwelling site Serteya II. *Archaeology of Lake Settlements IV-II Millennium BC: Chronology of Cultures and Natural-Climathic Rhythms*. Saint Petersburg: State Hermitage Museum. p 109–14.

Jaziehienko I, Jozwiak B. 2004. Chronologia poznych faz kultury dniepro-donieckiej na Bialorusi w swetle najnowszych oznaczen radioweglowych sa stanowiska Prorwa 2, rejon Rogaczowski. *Folia Praehistorica Poznaniensia* XII:47–64.

Kulkova MA, Savelieva LA. 2003. Vosstanovlenie paleoklimaticheskih uslovii golocena v raione zabolochennyh ozer v doline reki Serteiki v Velizhskom raione Smolenskoi oblasti. *Drevnosti Podvin'ya: istoricheskii aspekt* [The reconstruction of paleoclimatic conditions of Holocene in the bog region of Serteyka river in Velizhsky area of Smolensk oblast' (Antiquity of Podviniya: historical aspect)]. Saint Petersburg: Izdatelstvo Gosudarstvennogo Jermitaja. p 291–9.

Mazurkevich A, Dolbunova E. 2011. Underwater investigations in Northwest Russia: archaeology of pile-dwellings. In: Benjamin J, Bonsall C, Pickard C, Fischer A, editors. *Underwater Archaeology and the Submerged Prehistory of Europe*. Oxford: Oxbow Books. p 158–72.

Mazurkevich AN, Dolukhanov PM, Shukurov A, Zaitseva GI. 2009. Mesolithic and Neolithic in the Western Dvina–Lovat area. In: Dolukhanov PM, Sarson GR, Shukurov AM, editors. *The East European Plain on the Eve of Agriculture*. BAR International Series 1964. Oxford: Archaeopress. p 145–53.

Mazurkevich A, Dolbunova E, Maigrot Y, Hookk D. 2010. Results of underwater excavations of Serteya II and research of pile-dwellings in Northwest Russia. *Archaeologia Baltica* 14:47–64.

Nakamura T, Taniguchi Y, Tsuji S, Oda H. 2001. Radiocarbon dating of charred residues on the earliest pottery in Japan. *Radiocarbon* 43(2B):1129–38.

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

Regert M. 2011. Analytical strategies for discriminating archaeological fatty substances from animal origin. *Mass Spectrometry Reviews* 30(2):177–220.

Regert M, Mirabaud S. 2014. Substances naturelles exploitées sur les sites de Chalain et Clairvaux: nature et fonction des matériaux organiques amorphes. In: Arbogast RM, editor. *Entre archéologie et écologie, une Préhistoire de tous les milieux. Mélanges offerts à Pierre Pétrequin*. Presses universitaires de Franche-Comté. p 79–91.

Regert M, Dudd SN, van Bergen PF, Pétrequin P, Evershed RP. 2001. Investigations of solvent extractable lipids and insoluble polymeric components: organic residues in Neolithic ceramic vessels from Chalain (Jura, France). *British Archaeological Reports* 939:78–90.

Sablin MV, Siromyatnikova EV. 2009. Animal remains from Neolithic sites in northwestern Russia. *The East European Plain on the Eve of Agriculture. BAR International Series 1964*. Oxford: Archaeopress. p 153–8.

Smits L, van der Plicht H. 2009. Mesolithic and Neolithic human remains in the Netherlands: physical anthropological and stable isotope investigations. *Journal of Archaeology in the Low Countries* 1:55–85.

Zaitseva GI, Vasil'ev SS, Dergachev VA, Mazurkevich AN, Semencov AA. 2003. *Novye issledovaniya pamyatnikov basseina Zapadnoi Dviny i Lovati: raspredelenie radiouglerodnyh dat, korrelyaciya s izmeneniem prirodnyh processov, primenenie matematicheskoi statistiki (Drevnosti Podvin'ya: istoricheskii aspect)* [New investigations of archaeological sites of Western Dvina and Lovat' basin: distribution of radiocarbon dates, correlation with changes of environmental processes, the application of mathematic statistics (Antiquity of Podviniya: historical aspect)]. Saint Petersburg: Izdatelstvo Gosudarstvennogo Jermitaja. p 140–54.

Zaitseva GI, Kulkova MA, Mazurkevich AN. 2014. Radiocarbon chronology of Neolithic of Dnepr-Dvina region. In: *Archaeology of Lake Settlements IV-II Millennium BC: Chronology of Cultures and Natural-Climatic Rhythms*. Saint Petersburg: Izdatelstvo Gosudarstvennogo Jermitaja. p 67–85.

Zvelebil M. 1996. The agricultural frontier and transition to farming in the circum-Baltic region. In: Harris DR, editor. *The Origins and Spread of Agriculture and Pastoralism in Eurasia*. London: UCL Press. p 323–45.