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AQUATIC RADIOCARBON RESERVOIR OFFSETS IN THE SOUTHEASTERN BALTIC

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ABSTRACT. The aim of this article is to discuss radiocarbon dating offsets due to freshwater and marine reservoir effects (FRE and MRE, respectively) in the southeastern Baltic. Thirty-six ^{14}C dates from Lithuanian coastal and inland Subneolithic, Neolithic, and Bronze Age sites as well as two Mesolithic-Neolithic cemeteries are presented here. Accelerator mass spectrometry (AMS) dates, sometimes paired or tripled, have been obtained on samples of various origin, foodcrusts, or visible charred deposits adhering to the surfaces of ceramic vessel walls were also dated and investigated for stable isotope signals. The results argue for a significant freshwater component in the food processed in ceramic vessels during the Subneolithic and Neolithic. Paired dating of ungulate and human bones at the Spiginas and Donkalnis cemeteries (6300–1900 cal BC) does not suggest an FRE, although stable isotope data on human bone collagen strongly suggest a large input of freshwater food in the diet. An FRE in the order of 320–510 yr was estimated for the Šventoji paleolagoon around 3000 cal BC. At the same time, the FRE of the Curonian Lagoon could be larger as implied by large apparent ^{14}C ages of modern pike-perch (981 ± 30 BP) and bream (738 ± 30 BP) bones as well as “foodcrust” offsets (650–530 yr) at Nida (3500–2500 cal BC). An MRE of 190 ± 43 yr was estimated for the southeastern coast of the Littorina Sea according to offsets between dates of seal bones and terrestrial samples at Nida and Šventoji. Any FRE at Lake Kretuonas remains uncertain due to the limited work to date.

INTRODUCTION

In Europe, radiocarbon dating offsets due to freshwater reservoir effects (FRE) have been studied extensively for more than a decade (Cook et al. 2001; Fischer and Heinemeier 2003; Ascough et al. 2010; Keaveney and Reimer 2012; Philippsen and Heinemeier 2013). In the east Baltic, the problem was first recognized only in 2010 when surprisingly old ^{14}C dates were obtained on Subneolithic and Neolithic[1] foodcrusts or visible charred deposits adhering to the surfaces of ceramic vessel walls. At the time, an AMS dating program of 18 foodcrusts from various Subneolithic/Neolithic sites in Lithuania was initiated (Piličiauskas et al. 2011). Two main shortcomings prevented an adequate understanding and definition of the extent of the suspected aquatic reservoir offsets at that time. The first one was that context dates were absent or lacked precise spatial definition. Similarly, the foodcrust dates were mostly determined on samples from old excavations with insufficient contextual detail. Another issue in 2010 was the absence of any data on the composition of the foodcrusts, especially in detecting the presence of an aquatic food contribution to their formation. Both shortcomings have been tackled in the last 4 yr to a significant extent. From 2012–2014, new excavations at key coastal sites, i.e. Nida and Šventoji 4, have provided a range of well-documented terrestrial, marine, and freshwater samples. Moreover, paired dates from Mesolithic graves combined with C/N stable isotope data on bone collagen shed new light on the same issue from a different perspective. An FRE in the order of 800–900 yr has been recently estimated for Lake Burtnieki in Latvia adjacent to the largest Stone Age cemetery in the east Baltic (Meadows at al. 2014). This highlights the need to reconsider the existing chronologies for Stone Age cemeteries in the southeastern Baltic, e.g. Spiginas and Donkalnis in Lithuania, by exploring potential offsets between herbivore and human bone dates. Finally, a molecular and isotope study of Lithuanian foodcrusts and pottery sherds would provide the first evidence of food processed in the ceramic vessels. Therefore, the aim of this paper is to present and discuss new evidence on aquatic, i.e. freshwater as well as marine, ^{14}C reservoir offsets in Lithuania. This is of considerable importance to the prehistoric sequence in the eastern Baltic

1. Subneolithic in this paper refers to pottery-using hunter-gatherer communities in the period 5000/4000–3200/2900 cal BC in Lithuania. The Neolithic dates to 3200/2900–2000 cal BC and refers only to the Rzucewo, Globular Amphora, and Corded Ware cultures, which show clear signs of agricultural or mixed-type economies.

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where the chronology is not as well established as in other countries, e.g. in Denmark or Germany, where the impact of reservoir effects is better understood.

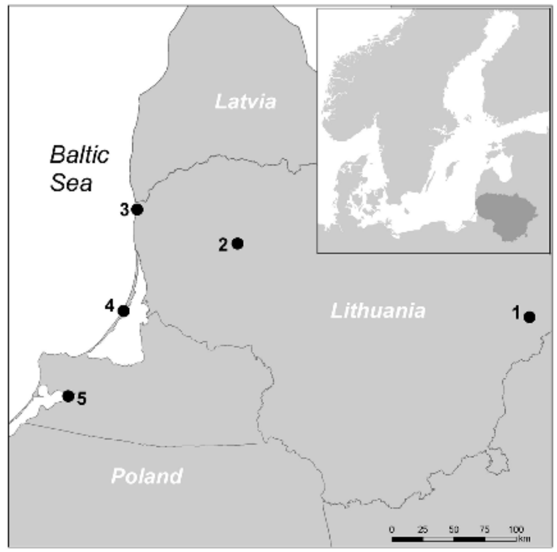

Figure 1 Sites investigated or mentioned: 1 – Lake Kretuonas (Kretuonas 1C, Žemaitiškė 2 and 3 sites); 2 – Lake Biržulis (Spiginas and Donkalnis cemeteries, Daktariškė 5 site); 3 – Šventoji paleolagoon (Šventoji 1–4, 6, 26, 47 sites); 4 – Nida site and Curonian Lagoon; 5 – Pribrezhnoye.

MATERIALS AND METHODS

As mentioned, 18 foodcrust AMS ^{14}C dates were available prior to the current phase of research (Piličiauskas et al. 2011). An additional nine foodcrusts from the coastal sites of Šventoji and Nida (Palanga city) as well as a single one from the inland Kretuonas 1C site (Švenčionys District) (Figure 1) were submitted for AMS dating to the Helsinki and Poznań ^{14}C laboratories. Charcoal embedded in the ceramic matrix and terrestrial plant remains (most probably lime bast) found in drilled repair holes as well as twisted around sherds as binding of net sinkers (Figure 2) were targeted along with other short-lived terrestrial materials found in a very close spatial association with ceramics or fish bones. This provided the opportunity for six paired dates associated with ceramics. Well-stratified and ^{14}C-dated profiles were also thoroughly studied for anomalous ^{14}C dates, e.g. at Nida.

An attempt was made to evaluate the impact of an FRE on the chronologies of the Mesolithic-Neolithic cemeteries at Spiginas and Donkalnis (Telšiai District, central Lithuania). Ungulate bones as well as tooth pendants were recovered from the same graves as human remains, providing ideal samples for paired dating. A single elk tooth pendant was dated in the Donkalnis 4 grave because an AMS date was already available on human bone from this context (Bronk Ramsey et al. 2000). The original human bone ^{14}C date for the Spiginas 1 grave had a very large uncertainty of 200 yr (Butrimas 1992). Thus, both human bone and ungulate bone fragments were dated from this context.

Thirty-six AMS dates are presented in this study. All wood or charcoal samples were pretreated using the acid-alkali-acid (AAA) method as described by Brock et al. (2010). For the bones, extraction of collagen was performed using the procedures originally described by Longin (1971), with further modifications (Piotrowska and Goslar 2002). The extracted collagen was ultrafiltered using precleaned Vivaspin™ 15 MWCO 30kD filters (Bronk Ramsey et al. 2004). Several samples had already been conserved with polymers [e.g. poly(butyl methacrylate)] (see Table 3). These were subject to careful precleaning with acetone and propanol before all other steps. Modern fish bones were boiled for 5 min, dried, chemically pretreated, and dated according to the described methodology.

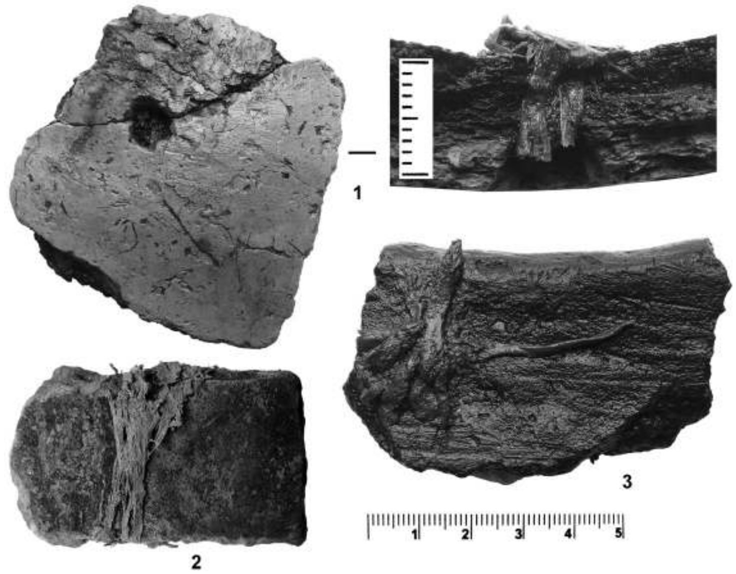

Figure 2 Subneolithic sherds from Šventoji 4 site with plant remains in repair holes or as binding of net sinkers.

Fifty-seven foodcrusts, 13 sherds with foodcrusts, and 10 sherds without visible deposits have been studied using lipid biomarker analysis, and bulk and compound-specific stable carbon isotope analysis (Heron et al. 2015). Those related to ^{14}C-dated foodcrusts are presented here because of their relevance to the current research problem. Separate samples of the foodcrusts were analyzed by bulk carbon and nitrogen isotope analysis without pretreatment. Each sample (~1 mg) was weighed in duplicate into tin capsules and analyzed using an elemental analyzer linked to a PDZ Europa 20/20 mass spectrometer. $\delta^{13}C$ and $\delta^{15}N$ measurements were determined relative to the VPDB and AIR international standards, respectively. Each element was analyzed separately, as the amount of C in the crusts was often far in excess of the amount of N. The instrument precision on repeated measurements was 0.3‰.The results are presented and discussed separately in the following subsections for each water basin in question, i.e. Lake Kretuonas, Lake Biržulis, the Šventoji paleolagoon, the Littorina Sea, and the Curonian Lagoon.

Lake Kretuonas

Lake Kretuonas (Švenčionys District, NE Lithuania) occupies an area of 861 ha. It is a shallow glacial lake with an average depth of 5.2 m connected to neighboring lakes by small streams. The bedrock is carbonaceous glaciofluvial sand and glacial till from the Weichselian glaciation and is the main source of dead carbon entering the lake in groundwater. The precipitation of carbonates on the lakebed was common throughout the Holocene (Kabailienė 2006). In 2007, the alkalinity of Lake Kretuonas was reported as 165 mg/L $CaCO_3$ equivalent (Database 2007).

The possibility of a substantial FRE at Lake Kretuonas was first raised when foodcrust dates suggested that textile-like impressed pottery is ~1000 yr older than had been previously assumed (Piličiauskas et al. 2011). For this study, only a single AMS date was obtained on a foodcrust sample from the Kretuonas 1C site. In addition, two foodcrust dates from the Žemaitiškė 2 and 3 sites were available for the same region. All three dated foodcrusts have been studied using bulk stable carbon and nitrogen isotope analysis. The results are presented in Table 1.

Table 1 ^{14}C dates and $\delta^{13}C$ and $\delta^{15}N$ values from Lake Kretuonas presented in or used for this study.

Site, year	Sample	Lab code	Date BP	Pottery type	%N	%C	C/N	$\delta^{15}N$ (‰)	$\delta^{13}C$ (‰)	References
Kretuonas 1C, 1987	foodcrust, sherd NM848:487/2408	Hela-2730	4608 ± 36	Late Narva	3.6	24.9	8.0	10.9	–30.5	this study
Žemaitiškė 2, 1980	foodcrust, sherd EM2430:513	Hela-2470	4351 ± 32	Textile-like impressed	4.4	40.3	10.8	10.3	–28.2	Piličiauskas et al. 2011; this study
Žemaitiškė 3, 1984	foodcrust, sherd K-31	Hela-2466	5319 ± 35	Textile-like impressed	6.0	35.5	6.9	11.4	–31	Piličiauskas et al. 2011; this study

Many prehistoric sites have been extensively investigated on the shore of the lake. Kretuonas 1C is a single-period and relatively short-lived site dating to the Early Bronze Age (Brazaitis 2002). Nine ^{14}C dates were available from an earlier study (Daugnora and Girininkas 2009). This includes a pike bone that did not demonstrate any significant offset compared to dates made on terrestrial animal bone. However, a foodcrust removed from a so-called Late Narva style vessel gave an offset of ~1000 yr (Figure 3; Table 1). At first glance, the foodcrust date (Hela-2730) appears to be an outlier. However, it is also possible that the pike bone date (Ki-11042) is the outlier, while the foodcrust date may be greatly affected by an FRE. Two attempts to redate pike bones from Kretuonas 1C failed in 2014 due to poor collagen preservation (<0.5% N and 5.0% C). The redating of Lithuanian Stone Age burials in 2014 revealed that many of them showed significant differences between the dates obtained by liquid scintillation in Kiev or Vilnius and by AMS at the Poznań laboratory (i.e. offsets of up to 1350 ± 75; Piličiauskas, unpublished data). In such cases, the condition of the bone and the chemical pretreatment protocol applied may explain the discrepancies between laboratories; for example, in bones with very little collagen, simply demineralizing a bone in acid may serve to concentrate any organic contaminants in the insoluble fraction. Unfortunately, details of the bone pretreatment protocol applied at the Kiev laboratory are not given (Kovaliukh and Skripkin 1994).

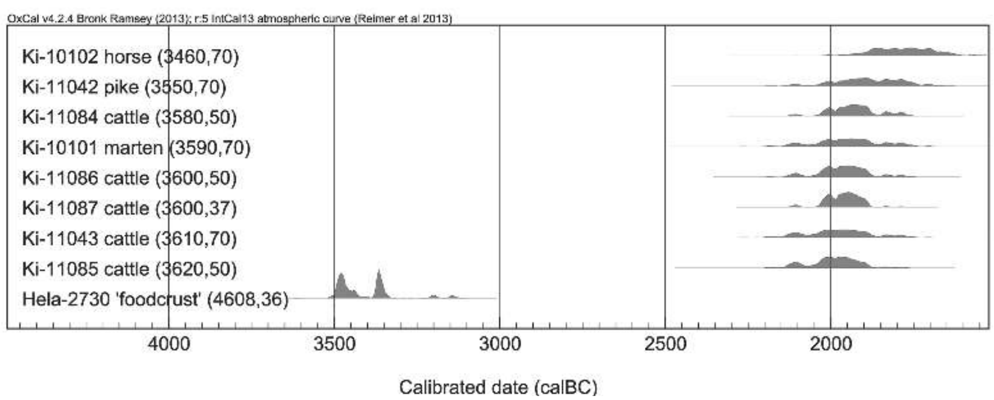

Figure 3 Calibration plot for the Kretuonas 1C site. Dates from the Kiev laboratory have been used from Daugnora and Girininkas (2009).

The bulk stable isotope values ($\delta^{15}N$ 10.9‰; $\delta^{13}C$ –30.5‰) of the dated foodcrust suggest a contribution of freshwater food and the potential for an FRE. Although lipid biomarker and compound-specific carbon isotope data have not been investigated for this sample, analysis of foodcrusts from the western Baltic (Craig et al. 2007, 2011) and from Nida (Heron et al. 2015) demonstrate a clear correlation between low $\delta^{13}C$ and high $\delta^{15}N$ values and the presence of aquatic lipid biomarkers such as isoprenoid fatty acids and long-chain (>C18) ω-(*o*-alkylphenyl)alkanoic acids. At this stage, it is not possible, without paired dates, to confirm an FRE from the small number of dates from Kretuonas 1C.

Lake Biržulis

Lake Biržulis (Telšiai District, W Lithuania) today occupies an area of 114 ha. However, it has shrunk significantly since a drainage program was started in 1954. It is a relict of a large glacial lake and is situated between moraine hills, connected to neighboring lakes by small streams. The bedrock is carbonaceous glaciofluvial sand and glacial till, which contains Ordovician-Silurian limestone transported by the ice sheet from Estonia during the Weichselian glaciation (Stančikaitė et al. 2004). In 2012, the alkalinity of Lake Biržulis was reported as 164 mg/L $CaCO_3$ equivalent (Database 2012a). Lake Biržulis is well known in the eastern Baltic because of the small Mesolithic-Neolithic cemeteries, Donkalnis and Spiginas, situated on former islands (Butrimas et al. 1985; Butrimas 1992).

In 2014, three AMS dates were obtained on ungulate bone, human bone, and an elk tooth from Spiginas and Donkalnis. Together with previously published dates, two pairs of human and ungulate bones/teeth deposited within the same graves became available. In addition, four dated foodcrusts have been studied using bulk stable carbon and nitrogen isotope analysis. The results are presented in Table 2.

Paired dates made on human bones and an ungulate tooth and bone from the same graves provide an opportunity to learn about any FRE at ancient Lake Biržulis (Table 2). The date 5020 ± 200 BP (Gin-5569) for the Spiginas 1 grave should be considered unreliable due to the very large error associated with it. A slight age difference of 100 ± 55 yr was observed between human and animal bone at Spiginas. However, the ^{14}C ages are not significantly different (Poz-61572: 5470 ± 40 BP and Poz-61569: 5370 ± 40 BP; $T = 3.1$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). The Donkalnis grave 4 gave an offset of 40 ± 75 yr between the human and terrestrial animal bones, which is not statistically significant (OxA-5924: 6995 ± 65 and Poz-61575: 6960 ± 40 BP; $T = 0.2$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). The negative $\delta^{13}C$ values (–23.3‰ and –22.8‰) combined with elevated $\delta^{15}N$ values (11.8‰ and 12.5‰) in the human bone collagen suggest a freshwater component to the diet of both individuals (Antanaitis-Jacobs et al. 2009). Theoretically, ungulates can also incorporate freshwater food into their diets; therefore, their bones could be influenced by an FRE. No stable isotope data for the animals at Lake Biržulis are available. However, at Šventoji, elk, red deer, and aurochs have very similar stable isotope signals to beavers ($\delta^{15}N$ 3.1‰ to 5.7‰; $\delta^{13}C$ –24.1‰ to –22‰; Antanaitis-Jacobs et al. 2009; Piličiauskas, unpublished data). The data support a minor FRE only at Lake Biržulis and the results of paired dates from the graves suggest that dates made on human bones at the Spiginas and Donkalnis cemeteries should be recognized as reliable. Foodcrust dates from Daktariškė 5, which is situated on the shore of Lake Biržulis, might be also reliable and may not need correction (Table 2; Piličiauskas et al. 2011), although the spatial variability of the FRE is unknown.

Šventoji Paleolagoon

The ancient Šventoji paleolagoon (Palanga, NW Lithuania) was situated at the southern end of a large fresh and brackish water system, consisting of shallow lakebeds interconnected by narrow channels including the deltas of several rivers. This lagoonal ecosystem emerged on a terrace of the retreating Littorina Sea around 4000 cal BC. By 2000 cal BC, it had become overgrown and drained due to isostatic land uplift (Piličiauskas et al. 2015). The Šventoji paleolagoon was shallow, muddy, warm, and highly productive, with many fishing stations and dwelling sites established by

Table 2 ^{14}C dates and $\delta^{13}C$ and $\delta^{15}N$ values from Lake Biržulis presented in or used for this study. Paired dates are shaded.

Site, year	Sample	Lab code	Date BP	Pottery type	%N	%C	C/N	$\delta^{15}N$ (‰)	$\delta^{13}C$ (‰)	References
Spiginas cemetery, grave 1, 1986	human bone, male, 35–44 yr	Gin-5569	5020 ± 200		—	—	3.6	11.8	–23.3	Butrimas 1992; Antanaitis-Jacobs et al. 2009
	human femur diaphysis, male, 35–44 yr	Poz-61572	5470 ± 40		—	—	—	—	—	this study
	ungulate long bone	Poz-61569	5370 ± 40		—	—	—	—	—	this study
Daktariškė 5, 1987	foodcrust, ? (zigzag ornamented)	Hela-2472	4370 ± 32	Globular Amphora	5.7	45.0	9.2	7.0	–28.1	Piličiauskas et al. 2011; this study
	foodcrust, sherd EM2245:750	Hela-2473	4661 ± 32	Narva	6.8	43.7	7.5	9.5	–28.6	Piličiauskas et al. 2011; this study
	foodcrust, sherd EM2245:2569	Hela-2471	5115 ± 34	Comb-like	8.8	47.8	6.3	9.6	–28.7	Piličiauskas et al. 2011; this study
	foodcrust, sherd D/5/9 6A	Hela-2599	4862 ± 36	Textile-like impressed	7.7	48.8	7.4	6.6	–30	Piličiauskas et al. 2011; this study
Donkalnis cemetery, grave 4, 1982	human bone, male, 50–55 yr	OxA-5924	6995 ± 65		—	—	3.6	12.5	–22.8	Bronk Ramsey et al. 2000; Antanaitis-Jacobs et al. 2009
	elk tooth	Poz-61575	6960 ± 40		—	—	—	—	—	this study

Subneolithic and Neolithic people (Piličiauskas et al. 2012). The nearest source of dead carbon was carbonaceous sand of the Baltic Ice Lake. However, the sand was isolated from the bottom of the lagoon by 0.5–2 m of carbonate-free Littorina Sea sand. The lagoon and later lagoonal lake was fed with freshwater by the Šventoji River, 68 km in length. It has a shallow valley and derives water almost exclusively from precipitation (Gailiušis et al. 2001). In 2012, the alkalinity of the Šventoji River was reported as 193.5 mg/L $CaCO_3$ equivalent (Database 2012b).

Šventoji 4 is a key site for studying aquatic reservoir effects because of the excellent preservation of artifacts and ecofacts including organic materials within the freshwater gyttja. It was excavated extensively between 1967 and 2006 (Juodagalvis and Simpson 2000; Rimantienė 2005). However, these materials were not documented properly or remain undated. In 2014, excavations at Šventoji 4 were resumed by investigating a 2 × 18 m trench and dating 20 samples by AMS (Piličiauskas, in press).

A total of 13 AMS dates were made on various materials (foodcrust, lime bast, wood, charcoal, fish bone, burnt water chestnut, and hazelnut) from Šventoji 4, 6, and 47 are presented in this study. Paired or sometimes tripled samples have been dated from the same sherd or from the well-documented stratified sedimentary sequence at Šventoji 4. In addition, nine dated foodcrusts have been studied using bulk stable carbon and nitrogen isotope analysis. The results are presented in Table 3.

Possible FREs were explored by dating a pike mandible, a small twig (*Salix*), and a charred water chestnut (*Trapa natans*) fruit, which were all found in very close spatial association: a thin lens of sand within the gyttja layer (Figure 4). No statistically significant ^{14}C age difference was found between the twig and water chestnut ages (Poz-64694: 4365 ± 30 BP and Poz-64696: 4350 ± 30 BP). The water chestnut does not appear to exhibit an FRE. In contrast, an offset of 320 ± 42 yr is evident for the pike mandible (Poz-64697: 4685 ± 30 BP) relative to the date of the twig (Poz-64694: 4365 ± 30 BP). These ^{14}C ages are significantly different ($T = 77.5$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). This offset cannot be recognized as the actual FRE for the former lagoon at Šventoji because of the variation within and between fish species that has been documented in previous studies on FREs in other areas (e.g. Keaveney and Reimer 2012; Fernandes et al. 2013). Another ^{14}C date determined on a fish bone from the earlier excavations gave a date of 4875 ± 65 BP (Tua-2076; Juodagalvis and Simpson 2000). Unfortunately, information on the precise fish species dated in Trondheim/Uppsala is not available, although it is known that these bones were found in the same lowermost horizon excavated in 2014. An offset of 510 ± 72 yr can be estimated in this case (i.e. the difference between 4365 ± 30 and 4875 ± 65 BP). The offsets 320 ± 42 and 510 ± 72 yr are significantly different ($T = 5.3$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978), and this indicates intraspecies variation of FRE or that dated fishes are not of the same calendar age.

The dates on the foodcrusts are in line with the findings described earlier. Age differences between foodcrusts and terrestrial materials are less than 510 ± 72 and 320 ± 42 yr at the Šventoji Subneolithic and Neolithic sites, respectively. A first pair of dates is from the Šventoji 4 site. A Subneolithic foodcrust (Poz-61563: 4500 ± 60 BP) and associated lime bast found in a drilled repair or binding hole (Poz-61564: 4265 ± 35 BP) revealed an offset of 240 ± 69 yr. The dates are statistically significantly different ($T = 11.6$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). Another pair comprising foodcrust and terrestrial plant remains at the same site demonstrated an offset of 300 ± 49 yr (Poz-66914: 4495 ± 35 BP and Poz-66913: 4200 ± 35 BP). The dates are significantly different ($T = 35.5$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). The last pair of dates was obtained on a Subneolithic sherd at the Šventoji 6 site situated on the same lake. The foodcrust (Poz-61585: 4520 ± 35 BP) appears older than a fragment of charcoal (Poz-61586: 4440 ± 35 BP) embedded within the ceramic

Table 3 ^{14}C dates and $\delta^{13}C$ and $\delta^{15}N$ values from the Šventoji paleolagoon presented in or used in this study. Paired dates are shaded. [1]sherds treated with polymers.

Site, year	Sample	Lab code	Date BP	Pottery type	%N	%C	C/N	$\delta^{15}N$	$\delta^{13}C$	References
Šventoji 1, 2006	foodcrust, test-pit 95	Hela-2476	4625 ± 32	Globular Amphora	6.2	42.0	7.9	10.0	–26.8	Piličiauskas et al. 2011; this study
Šventoji 2, 1966	foodcrust, sherd EM2135:141	Hela-2477	4507 ± 32	Globular Amphora	3.2	49.1	18.1	9.8	–26.1	Piličiauskas et al. 2011; this study
Šventoji 3, 2005	foodcrust, sherd ASAS 114 (08.)	Hela-2462	4756 ± 32	Narva	7.0	50.7	8.5	11.1	–26.2	Piličiauskas et al. 2011; this study
Šventoji 3, 2005	foodcrust, sherd ASAS 85 (07.)	Hela-2465	4783 ± 32	Narva	7.5	47.5	7.4	11.1	–26.7	Piličiauskas et al. 2011; this study
Šventoji 3, 2005	foodcrust, sherd ASAS 31 (08.17)	Hela-2461	4827 ± 33	Narva						Piličiauskas et al. 2011
Šventoji 4, 2006	plant remains, sherd 34[1]	Poz-61564	4265 ± 35							this study
	foodcrust, sherd 34[1]	Poz-61563	4500 ± 60	Narva	6.1	45.6	8.7	10.2	–26.7	this study
Šventoji 4, 2003	foodcrust, 1436 (09.14)	Hela-2464	4805 ± 33	Narva	8.4	40.8	5.7	11.2	–27.2	Piličiauskas et al. 2011; this study
Šventoji 4, 2014	plant remains, sherd 1060	Poz-66913	4200 ± 35							this study
	foodcrust, sherd 1060	Poz-66914	4495 ± 35	Narva						this study
Šventoji 4, 1998	'freshwater' fish bone, bone lens	Tua-2075	4875 ± 65							Juodagalvis and Simpson 2000
Šventoji 4, 2014	Wood twig, bone lens, 1667	Poz-64694	4365 ± 30							this study
	water chestnut, bone lens, 1665	Poz-64696	4350 ± 30							this study
	pike bone, bone lens, 1667	Poz-64697	4685 ± 30							this study
Šventoji 4, 2014	hazelnut, 1242	Poz-66917	4160 ± 35							this study
Šventoji 4, 2014	hazelnut, 1244	Poz-66918	4140 ± 35							this study
Šventoji 6, 1983	charcoal, sherd 2138:1410[1]	Poz-61586	4440 ± 35							this study
	foodcrust, sherd 2138:1410[1]	Poz-61585	4520 ± 35	Narva	6.0	47.0	9.2	12.3	–26.9	this study
Šventoji 26, 2005	foodcrust, test-pit	Hela-2463	4835 ± 34	Combed-like	4.1	38.2	10.9	10.7	–27	Piličiauskas et al. 2011; this study
Šventoji 47, 2013-2014	foodcrust, sherd 285	Poz-65430	3660 ± 30	Post-corded	3.5	42.9	14.3	4.5	–25.9	this study
	wood twig	Poz-65429	3620 ± 30							this study

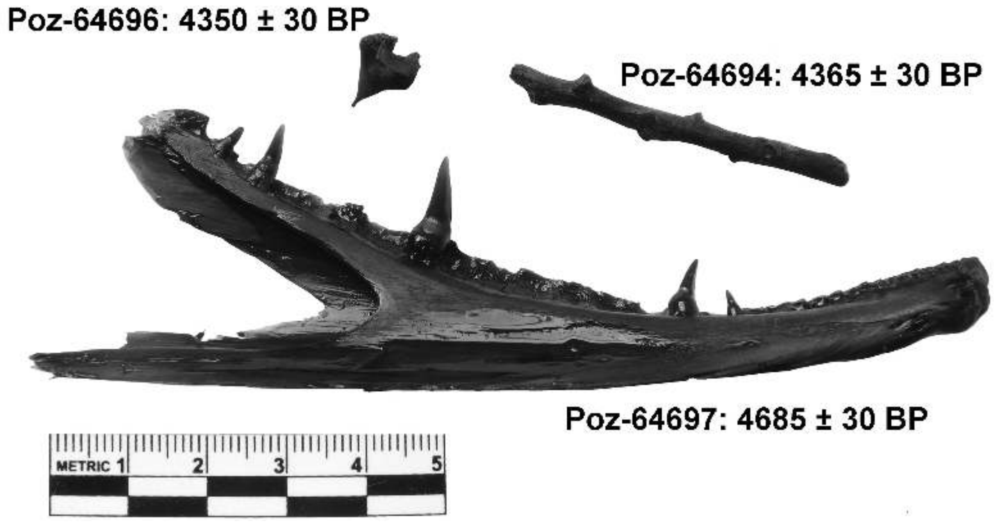

Figure 4 ^{14}C-dated water chestnut (*Trapa natans*), wood (*Salix*), and pike (*Esox lucius*) mandible from Šventoji 4 site

matrix by 80 ± 49 yr. This offset is not as evident as in the case with the cord remains, and the ^{14}C ages are not significantly different ($T = 2.6$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). This could be explained in several ways, i.e. by a variable FRE, by different composition of the food remains, or an "old wood" effect. Nevertheless, the foodcrust offsets fit well with the general pattern, i.e. they are lower than the FRE. This is understandable, as freshwater food may have been processed in the same pots with food from terrestrial and/or marine environments.

At Šventoji, a single case where an offset is not evident has been demonstrated by a foodcrust from Šventoji 47. It belongs to the transitional Neolithic–Early Bronze Age period, i.e. ~2000 cal BC. This is a time of marine regression when lagoonal lakes were transformed into swamps connected by small rivers. Post-corded type ceramics were found within the sandy peat of a river bed. The foodcrust gave a date of 3660 ± 30 BP (Poz-65430), while a small twig uncovered just a few centimeters from the potsherd on the same level was dated to 3620 ± 30 BP (Poz-65429). These ^{14}C ages are not significantly different ($T = 0.9$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). While the bulk isotope signal does not suggest aquatic resources (δ^{15}N 4.51‰; δ^{13}C –25.87‰), the presence of aquatic lipid biomarkers, namely 4,8,12-trimethyltridecanoic acid (TMTD) and C16, C18, and C20 ω-(*o*-alkylphenyl)alkanoic acids, does suggest at least a contribution from aquatic resources. The lower δ^{15}N and elevated δ^{13}C values greatly differentiate the Šventoji 47 foodcrust from Neolithic and Subneolithic foodcrusts found at the other Šventoji sites (Figure 5; Table 1). At Šventoji, the foodcrust dates cannot be corrected according to the composition of the food residue because it is not possible to determine quantitatively the contribution of marine, freshwater, and terrestrial carbon to the residues. Clearly, further work is required to resolve potential offsets and the presence of aquatic resources in pottery from transitional Neolithic–Early Bronze Age period ceramics.

Littorina Sea

Two AMS dates made on seal bones from Šventoji 4 and Nida are presented in Table 4. In general, marine reservoir effects (MREs) in the Baltic Sea are lower than those of the global ocean. Dates on seal bone from the Littorina Sea period in the SW Baltic gave offsets of about 300–250 yr (Andrén et al. 2000; Rundkvist et al. 2004). However, a different MRE might be expected for the eastern Baltic.

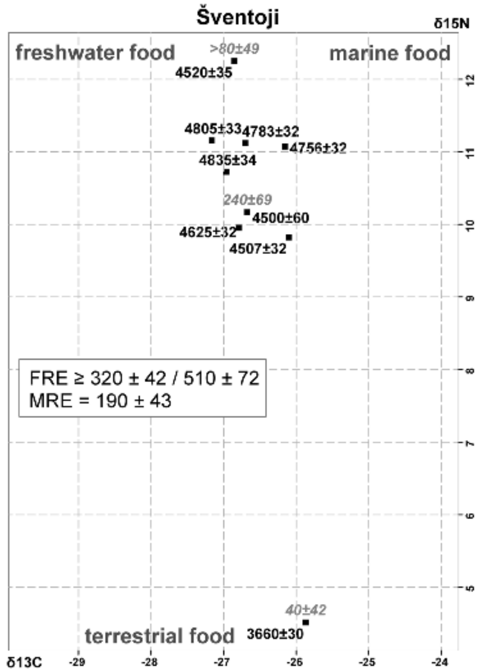

Figure 5 ^{14}C dates and offsets in a context of bulk stable isotope values of foodcrusts at the Šventoji sites (FRE = freshwater reservoir effect; MRE = marine reservoir effect).

Table 4 Seal bone ^{14}C dates from Šventoji 4 and Nida sites.

Site, year	Sample	Lab code	Date BP	References
Šventoji 4, 2014	seal bone, 1827	Poz-66915	4340 ± 35	this study
Nida, 2013	seal bone, 610	Poz-61587	4140 ± 35	this study

Both seal bones were not characteristic enough for species determination. At Nida, the seal bone date (Poz-61587: 4140 ± 35 BP) from the upper archaeological layer appears to be older by 100 ± 45 yr than a hazelnut shell date (Poz-61703: 4045 ± 30 BP) from the lower layer (Figure 6). If the seal bone is not older than sediment it was found within, then the MRE must be a minimum of 100 yr or larger. At Šventoji 4, the seal bone was dated to 4340 ± 35 BP (Poz-66915). This find was found within a horizon of gyttja that was dated by two hazelnuts dating to 4160 ± 35 BP (Poz-66917) and 4140 ± 35 BP (Poz-66918) (Figure 7). An offset between the combined date of the hazelnuts (4150 ± 25 BP) and the seal bone date (4340 ± 35 BP) is 190 ± 43 yr, which corresponds to statistically different ^{14}C ages ($T = 19.7$, $T'(5\%) = 3.8$, $\nu = 1$; Ward and Wilson 1978). This value may be considered as the MRE of the Littorina Sea, valid at least for the SE Baltic coast. Although the dated Šventoji 4 seal has stable isotope values (δ^{15}N 12.02‰; δ^{13}C –16.59‰) that are very close to harbor seal (*Phoca vitulina*) and ringed seal (*Phoca hispida*) values for Šventoji sites (see Antanaitis-Jacobs et al. 2009), without species identification we cannot rule out that the seal or what it ate migrated over significant distances, and the estimated MRE may therefore be applicable as an average over a wider area.

Curonian Lagoon

The Curonian Lagoon is the largest lagoon in Lithuania. Currently, it covers an area of 1584 km^2 with an average depth of 3.8 m. The lagoon appeared approximately at the same time as the Šventoji paleolagoon emerged, around 4000 cal BC, during the Littorina Sea regression. A narrow sandy spit isolated the lagoon from the Littorina Sea. The lagoon is supplied with water by the largest Lithuanian river, the Nemunas. The river runs in a deep valley and groundwater comprises roughly 35% of the entire water yield (Rainys 2009). Groundwater is usually enriched with dead carbon coming from carbonaceous glacial tills. In 2012, the alkalinity of the lower Nemunas was reported as 190 mg/L $CaCO_3$ equivalent (Database 2012b).

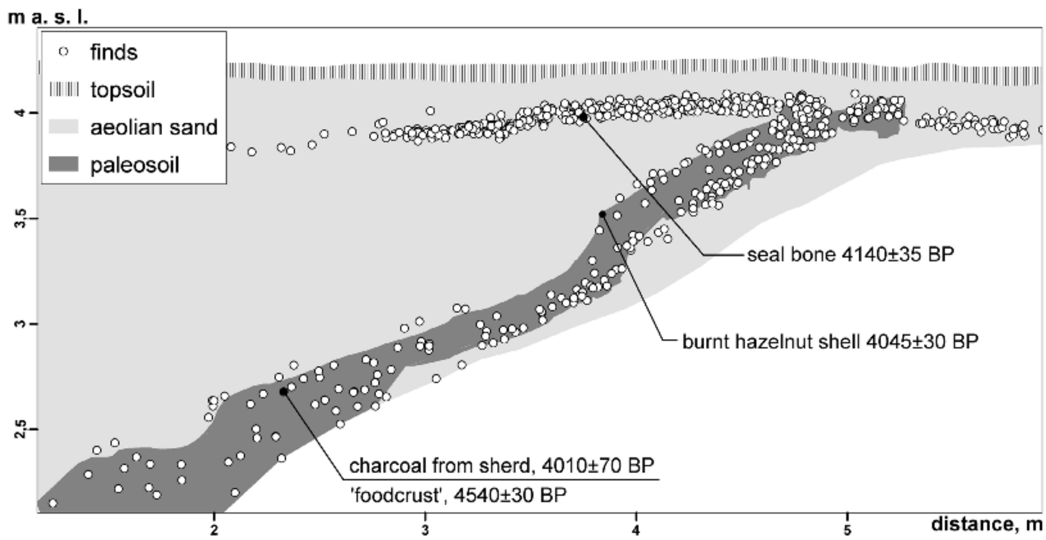

Figure 6 Stratigraphic profile with ^{14}C-dated Neolithic Rzucewo culture horizons at Nida (2012 research by G Piličiauskas)

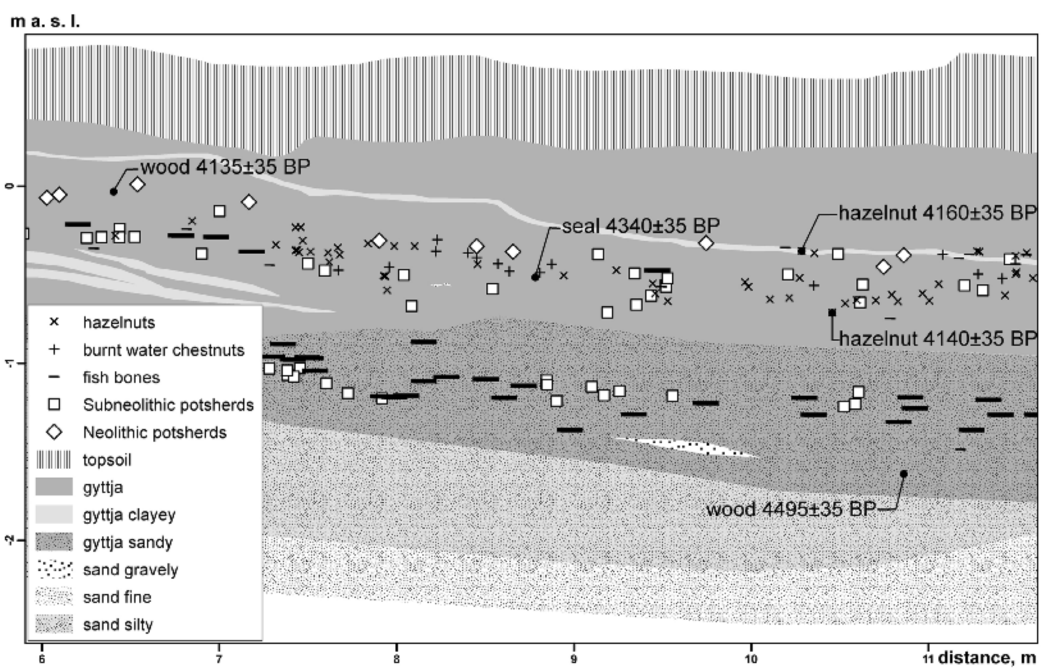

Figure 7 Stratigraphic profile at Šventoji 4 site with dated wooden artifacts, hazelnuts, and seal vertebra (2014 research by G Piličiauskas).

Many Neolithic Rzucewo culture (other names are Bay Coast culture, Pamariai culture, or Haffküstenkultur) sites are known on the spit. In contrast, the Subneolithic is represented only by a few sherds occasionally found on Neolithic sites (Rimantienė 1999; Piličiauskas 2013). The most intensively investigated site is Nida 1, with more than 100,000 sherds collected from the 1974–1978 excavations (Rimantienė 1989). Between 2011–2013, excavations were resumed by G Piličiauskas (in press). A total of 16 AMS ^{14}C dates made on various materials (foodcrust, charcoal, and bulk organics) from Nida are presented in this study. In addition, two AMS dates were made on modern fish bones, pike-perch (*Sander lucioperca*), and bream (*Abramis brama*), caught in the Curonian Lagoon in 2014. Seven dated foodcrusts have also been studied using bulk stable carbon and nitrogen isotope analysis. All results are presented in Table 5.

At Nida, only a single paired date was possible for evaluation. The foodcrust (Poz-64683: 4540 ± 30 BP) and charcoal (Poz-64682: 4010 ± 70 BP) from the same sherd gave a statistically significant ^{14}C age difference of 530 ± 76 yr (T = 46.2, T'(5%) = 3.8, ν = 1; Ward and Wilson 1978). The charcoal embedded in the ceramic matrix (Poz-64682: 4010 ± 70 BP) was not apparently influenced by the "old wood" effect because burnt hazelnut shell from the same horizon gave a very similar age (Poz-61703: 4045 ± 30 BP) (Figure 6).

Even larger offsets for foodcrusts were observed from well-stratified and documented eolian deposits. For example, charcoal, bulk organics from paleosoils, and foodcrusts have been dated from an ancient depression filled with sand laminated with several humus or peaty horizons (Figure 8). It is very likely that the dates of bulk organic samples from the lower part of the section were affected by penetrating tree roots seeking the organic-rich humus horizons. A charcoal sample from one ceramic may be too old (Poz-49781: 4420 ± 40 BP), possibly because it was incorporated into the clay before the manufacture of the vessel, i.e. within the clay source. A charcoal date (Poz-49776: 4290 ± 35 BP) from the lower part of the section, together with bulk soil date (Poz-49778: 4230 ± 35 BP) from the mid-section appear reasonable. It is very likely that the charcoal (Poz-49776:

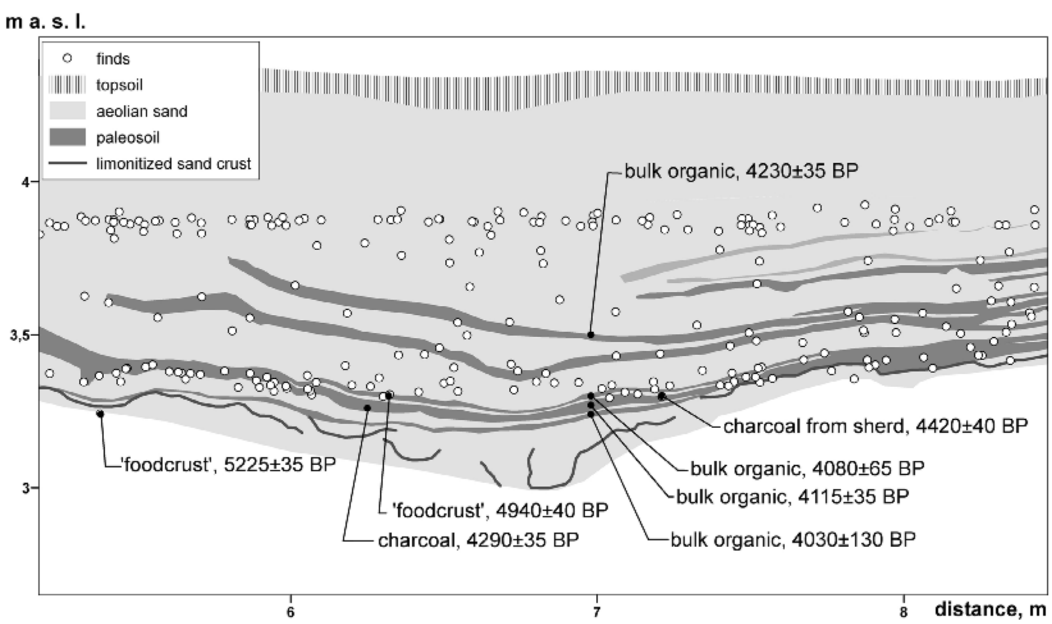

Figure 8 Stratigraphic profile with ^{14}C-dated Neolithic Rzucewo culture horizons at Nida (2012 research by G Piličiauskas)

Table 5 ^{14}C dates and $\delta^{13}C$ and $\delta^{15}N$ values from Nida and the Curonian Lagoon (modern fish) presented in or used for this study. Paired dates are shaded.

Sample, excavation year	Lab code	Date BP	Corrected age	Pottery type	%N	%C	C/N	$\delta^{15}N$	$\delta^{13}C$	References
foodcrust, sherd EM2243:2321, 1974	Hela-2468	4917 ± 34	4310 ± 56[1]	Rzucewo I	5.6	43.6	9.1	9.2	–29.3	Piličiauskas et al. 2011; this study
foodcrust, sherd EM2/10y, 1974	Hela-2474	5005 ± 34		Rzucewo I						Piličiauskas et al. 2011
foodcrust, EM2243:2087, 1974	Hela-2475	4850 ± 34		Rzucewo I						Piličiauskas et al. 2011
charcoal, area 2, fireplace 46, 1975	Vs-632	4460 ± 110								Rimantienė 1989
charcoal, fireplace 56, 1975	Bln-2592	4070 ± 50								Rimantienė 1989
wood, 1976	Vs-321	4630 ± 120								Rimantienė 1989
foodcrust, sherd EM2243:3778, 1976	Hela-2469	4946 ± 34	4340 ± 56[1]	Rzucewo I	4.3	34.6	9.4	8.8	–29.4	Piličiauskas et al. 2011; this study
foodcrust, sherd EM2243:3760, 1976	Hela-2728	4818 ± 43	4210 ± 62[1]	Rzucewo I	5.0	47.5	11.0	8.5	–31.6	this study
charcoal, area 1, fireplace 24, 1977	Vs-631	4620 ± 110								Rimantienė 1989
foodcrust, sherd EM2243:4331, 1977	Hela-2467	5041 ± 34	4430 ± 56[1]	Rzucewo I	6.5	30.8	5.6	9.6	–27.1	Piličiauskas et al. 2011; this study
foodcrust, sherd EM2243:5337, 1978	Hela-2729	5220 ± 40		Rzucewo I						this study
charcoal, sherd 121, 2011	Poz-49774	4620 ± 40		Narva						this study
foodcrust, sherd 189, 2011	Poz-49775	5225 ± 35	4610 ± 56[1]	Rzucewo I	4.2	36.1	10.0	9.8	–30.4	this study
bulk organics, trench 3, sample 29, 2012	Tln-3413	4080 ± 65								this study
bulk organics, trench 3, sample 32, 2012	Tln-3418	4030 ± 130								this study
bulk organics, trench 3, sample 18, 2012	Poz-49778	4230 ± 35								this study
bulk organics, trench 3, sample 30, 2012	Poz-49779	4115 ± 35								this study
charcoal, test-pit 8, 2012	Poz-65438	4630 ± 35								this study
charcoal, trench 3, B12/3, 2012	Poz-49776	4290 ± 35								this study
charcoal, test-pit 8, 2/4, 2012	Poz-49780	4210 ± 35								this study
charcoal, sherd 3366, 2012	Poz-49781	4420 ± 40								this study
foodcrust, sherd 1985 (3143), 2012	Poz-49783	4940 ± 40	4290 ± 66[2]	Rzucewo I	5.5	44.7	9.4	10.4	–30.7	this study
foodcrust, sherd 6921, 2013	Poz-64683	4540 ± 30	4010 ± 82[3]	Rzucewo II	5.5	36.6	7.8	8.3	–29	this study
charcoal, sherd 6921, 2013	Poz-64682	4010 ± 70		Rzucewo II						this study
hazelnut, A5/7, 2013	Poz-61703	4045 ± 30								this study
modern pike-perch, bone	Poz-66924	782 ± 30	981 ± 30[5]	caught in 10.2014						this study
modern bream, bone	Poz-68570	539 ± 30	738 ± 30[5]	caught in 10.2014						this study

[1] FRO (freshwater reservoir offset) corrected by 611 ± 44 yr; [2] FRO corrected by 650 ± 53 yr; [3] FRO corrected by 530 ± 76 yr; [4] MRO (marine reservoir offset) corrected by 190 ± 43 yr; [5] apparent ^{14}C age of modern sample when atmospheric ^{14}C concentration is 102.5 pMC.

4290 ± 35 BP) and Rzucewo dated foodcrust (Poz-49783: 4940 ± 40 BP) from the lowermost part of a sequence are coeval (Figure 8). Both samples were uncovered with only 4-cm difference in depth. As their locations in Figure 8 have been projected onto a profile from a 1-m-wide trench, they might originate from exactly the same horizon. Otherwise, their age differences should only be minimal because the intermediate layers of eolian sand could be formed over days or even hours. An offset of 650 ± 53 yr can be estimated between the charcoal (4290 ± 35 BP) and Rzucewo foodcrust (4940 ± 40 BP).

An offset of 650 ± 53 yr is unlikely to be a maximal limit at the Curonian Lagoon because of the complex nature of foodcrusts noted earlier. AMS dates of modern pike-perch and bream both caught in the lagoon in October 2014 gave conventional ^{14}C ages of 782 ± 30 BP (90.72 pMC) for pike-perch and 539 ± 30 BP (93.5 pMC) for bream. Assuming an atmospheric ^{14}C concentration in 2014 of 102.5 pMC (by extrapolation from Hua et al. 2013:Table S2a), the apparent ^{14}C ages were calculated as 738 ± 30 BP for bream and 981 ± 30 BP for pike-perch (Table 5). Considering a terrestrial food component in the diet of these fish, it is possible to assume that the modern FRE of the Curonian Lagoon is higher than the apparent ^{14}C ages of the fish and could be over 1000 yr. It is not possible to say that the same FRE existed during the Littorina Sea stage. However, the presence of a terrestrial component in the dated Nida foodcrusts will reduce the offset in these samples; therefore, an ancient FRE of similar magnitude to the modern offset is not unexpected.

Another important question is whether correcting dates made on other foodcrusts at Nida is possible. Consideration of the results of bulk stable isotope analysis performed on dated foodcrusts is important in this regard (Figure 9). The stable isotope values are rather dispersed. Although there is considerable variation in $\delta^{13}C$ in freshwater systems, the largest input of terrestrial food is to

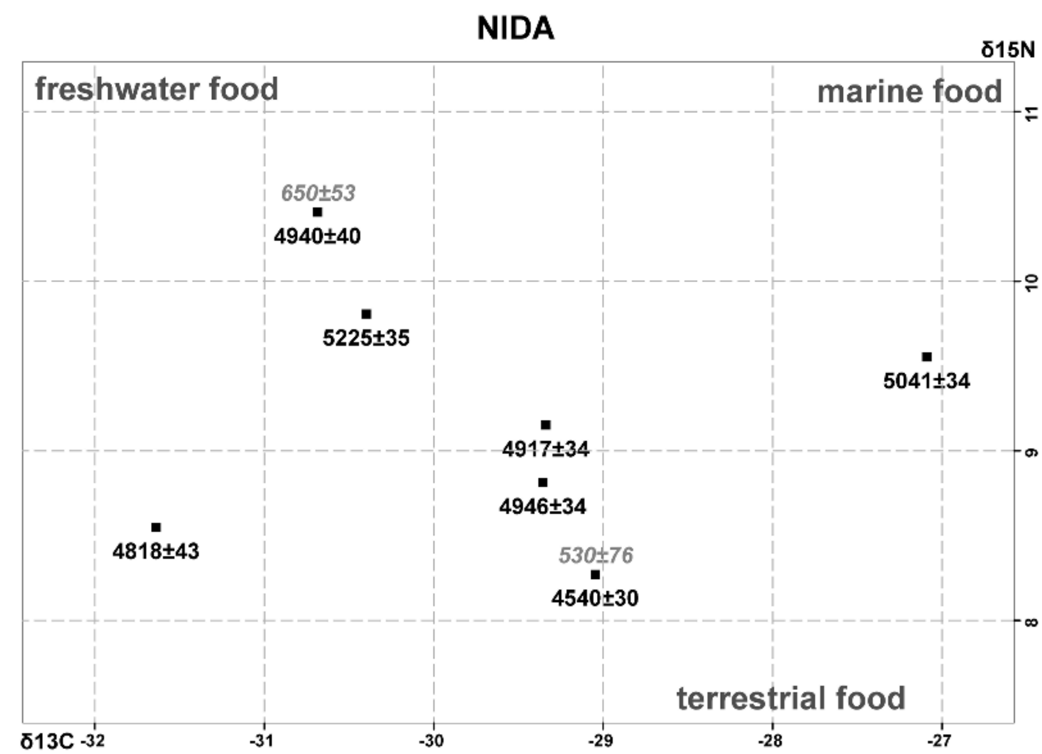

Figure 9 ^{14}C dates and offsets in the context of bulk stable isotope values of foodcrusts at Nida be expected in foodcrusts with less negative $\delta^{13}C$ values and low $\delta^{15}N$ values. This is supported by lipid biomarker and compound-specific carbon isotope analysis (Heron et al. 2015). The bulk isotope values for the foodcrust with an estimated FRO of 650 ± 53 yr are $\delta^{15}N$ 10.41‰ and $\delta^{13}C$ –30.69‰. One foodcrust with a FRO of 530 ± 76 yr has bulk isotope values of $\delta^{15}N$ 8.27‰; $\delta^{13}C$ –29.05‰. Other foodcrusts demonstrated intermediate $\delta^{15}N$ values in between 10.41‰ and 8.27‰. This indicates that the two foodcrusts for which FROs have been estimated might contain minimal and maximal amount of aquatic carbon, of course, only within a range of isotopically investigated and dated foodcrusts. That leads to the assumption that the majority of isotopically investigated and dated foodcrusts should have an offset between 650 ± 53 and 530 ± 76 yr (Figure 9). A mean value between those offsets is 611 ± 44 yr. This age difference was used to correct the other isotopically investigated foodcrust dates at Nida, apart from the two cases where individual corrections were possible (i.e. cases with foodcrust dates Poz-64683: 4540 ± 30 BP and Poz-49783: 4940 ± 40 BP). Without paired dates or dated contexts, foodcrust dates cannot be corrected individually, however. A small number of known offsets do not allow us to estimate a quantitative dependence between offset size and stable isotope data.

What do the corrected foodcrust dates tell us about the chronology of Nida and the Rzucewo culture (Figure 10)? Even after correction, the main conclusion presented by Piličiauskas et al. (2011) remains valid: the Rzucewo culture appeared in the SE Baltic in the late 4th millennium cal BC, i.e. several hundred years prior to the Corded Ware or Battle Axe phenomena in central and northern Europe. To be more specific, the Rzucewo culture starts ~3200 cal BC, while the earliest Corded Ware culture graves appear only around 2900/2700 cal BC in the southeastern Baltic (Antanaitis-Jacobs et al. 2009; Piličiauskas, in press). The Rzucewo culture continued to exist into the mid-

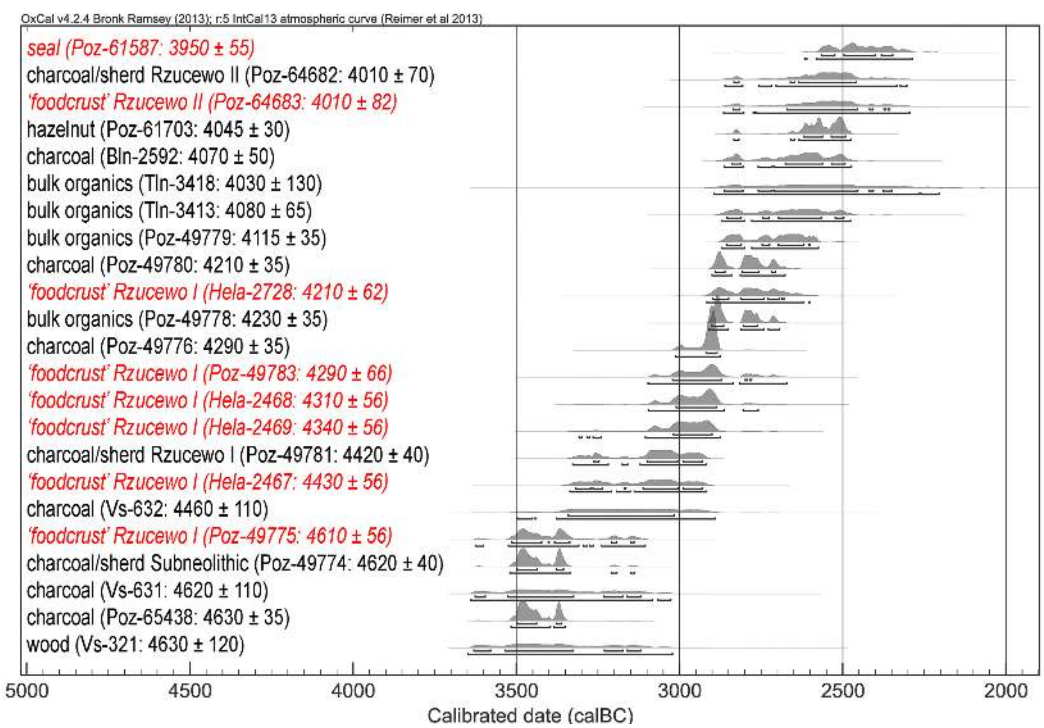

Figure 10 Calibration plot for Nida. ¹⁴C ages corrected for MRE and FROs are marked with red color and italic font. The earliest and latest dates (Le-1361: 11090 ± 100; Vs-320: 3470 ± 70; Le-1384: 3190 ± 60; Le-1976: 2850 ± 40), are inconsistent with archaeological and geological data, and are not included.

dle part of the Neolithic (2900/2700–2500 cal BC), i.e. the Corded Ware period (Rzucewo II). S-profile thin-walled beakers became common in the late stage at the same time as the prolonged bowls/lamps disappeared. The idea of the beginning of the Rzucewo culture in the 4th millennium cal BC is incompatible with the traditional understanding of that culture as an outcome of a so-called pan-European horizon (e.g. Rimantienė 1984; Machnik 1997). However, it is in good agreement with very old ^{14}C dates obtained from the Pribrezhnoye site, Kaliningrad oblast, Russian Federation. Here, pottery with elaborated "corded" designs was found within slightly deepened long houses. Ten ^{14}C dates belong to the period 3500–3000 cal BC, with only two dates in the period 3000–2700 cal BC. All dates were made on charcoal and wood, thus excluding any FRE (Saltsman 2010).

FINAL REMARKS

Recently published studies on FRE have emphasized its complexity and variability in space and time (e.g. Ascough et al. 2010; Fernandes et al. 2012; Philippsen and Heinemeier 2013). Thus, the offsets presented and discussed herein should not be used directly when discussing other aquatic systems and periods. Despite these and other shortcomings, the documentation of aquatic reservoir offsets may bring many scientific benefits. ^{14}C offsets can provide us with data on the nature of food consumed by prehistoric people with offsets used as proxy data in paleodiet studies. The question of primary importance is to determine accurate ages of artifacts or ecofacts. The easiest way to avoid the possibility of FRE and MRE is not to date freshwater and marine samples. However, this option is not always available. Sometimes, there is little or no choice; therefore, knowledge of possible offsets becomes highly valuable. Furthermore, a large number of dates on foodcrusts are already available. It would be preferable to correct and use them in future research, rather than reject them outright. Finally, continued dating of foodcrusts combined with molecular and isotopic analysis of lipid residues can help to select samples with terrestrial signals, thus avoiding potential reservoir effects.

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