Radiocarbon, Vol 60, Nr 5, 2018, p 1597–1610 DOI:10.1017/RDC.2018.95 Selected Papers from the 2nd International Radiocarbon and Diet Conference: Aquatic Food Resources and Reservoir Effects, 20–23 June 2017, Aarhus, Denmark © 2018 by the Arizona Board of Regents on behalf of the University of Arizona

DIET AND CHRONOLOGY OF NEOLITHIC-ENEOLITHIC CULTURES (FROM 6500 TO 4700 CAL BC) IN THE LOWER VOLGA BASIN

A Vybornov¹ • M Kulkova^{2*} • P Kosintsev³ • V Platonov⁴ • S Platonova⁴ • B Philippsen⁵ • L Nesterova²

¹Samara State University of Social Sciences and Education – History and Archaeology, Samara, Russia.

²Herzen State University – Geology and Geoecology, St.Petersburg, Russia.

³Institute of Plant & Animal Ecology – Mammals, Yekaterinburg, Russia.

⁴Samarskij nacional'nyj issledovatel'skij universitet imeni akademika S P Koroleva Ringgold standard institution – Chemistry, Samara, Samarskaa oblast', Russia.

⁵Aarhus University – Aarhus AMS Centre, Department of Physics and Astronomy, Ny Munkegade 120, Aarhus 8000, Denmark.

ABSTRACT. During the last several years, new multi- and single-layered archaeological sites, in which the most ancient Neolithic pottery in the Eastern Europe had been found, were excavated in the region of Lower Volga. Animal bones and organic materials were sampled from these sites for radiocarbon (14 C) dating and diet investigations. The evidence from these studies suggests that the first domestic animals in the Lower Volga region appeared in the Cis-Caspian culture of the Early Eneolithic. Lipid analysis of food crusts from pottery allowed the cooked food to be characterized. The detailed chronology from Neolithic (6500–5400 cal BC) to Eneolithic (5300–4700 cal BC) cultures, as well as the diet of these ancient people, were reconstructed.

KEYWORDS: animal bone, archaeozoology, dating, domestication, radiocarbon dating.

INTRODUCTION

Archaeological sites containing the most ancient pottery of the Lower Volga region have been investigated since the mid-1970s (Melentiev 1975, 1976; Vasiliev et al. 1989; Grechkina et al. 2014; Vybornov et al. 2015, 2016a, 2017). In 1980, the expedition of Samara Pedagogical University under the guidance of I.B. Vasiliev began the research of Neolithic and Eneolithic sites in the Northern Caspian region. During excavations, artifacts belonging to different periods and different cultural traditions were discovered. However, animal bones were not found in single-layered sites, so the Neolithic economy remained unknown. In the 1980s, several sites containing faunal remains were studied (Koltsov 2004; Yudin 2004). Nevertheless, it was difficult to distinguish between domestic and wild animal species. During these investigations, 12 radiocarbon (¹⁴C) dates from four archaeological sites were obtained. These dates are debatable (Vasiliev et al. 1989; Grechkina et al. 2014; Vybornov et al. 2015, 2016a, 2017), so it remained impossible to determine the chronology of the Neolithic-Eneolithic in this region. In the period from 2007 to 2017, new multi- and single-layered archaeological sites were excavated (Figure 1). The cultural layers contained a lot of animal bones. We were able to obtain large series of ¹⁴C dates on charcoal, animal bones, and food crusts for different sites. The results allow us to develop a detailed chronological schema for Neolithic-Eneolithic cultures of this region. The detailed characteristics of Eneolithic cultures of the Northern Cis-Caspian and the steppe Volga region were presented in the book of David Anthony (2007). Therefore, we will not discuss them further in this paper.

METHODS AND MATERIALS

Bones from cultural layers of archaeological sites were studied by palaeozoological analysis (Tables 1 and 2). Determinations of animal species from sites of Tenteksor I, Kurpezhe-Molla, and Karakhuduk I have been published by Kuzmina (1988). One of the authors (P Kosintsev) of our new study has studied these bone assemblages further. The results obtained (Tables 1

^{*}Corresponding author. Email: kulkova@mail.ru.



Figure 1 The map of Neolithic-Eneolithic sites in the Lower Volga basin.

and 2) show the results of this reinvestigation of species composition and distribution of different animal bones on these sites.

For ¹⁴C analysis, samples of charcoal and food crusts were pretreated with diluted HCl acid (1.2N) and NaOH (0.1N) to remove contaminants such as carbonates and organic acids from the soil originating from decomposed organic matter. The CO₂ produced from samples was graphitized before being dated by accelerator mass spectrometry (AMS) (Cottereau et al. 2007). For ¹⁴C dating of bones, collagen extraction was carried out according to the standard procedures of Arslanov and Svezhentsev (1993). For liquid scintillation counting (LSC), benzene cocktails were measured on a Quantulus 1220 liquid scintillation counting system. The ¹⁴C calibration program OxCal 4.3 was used for calibration of ¹⁴C dates (Bronk Ramsey et al. 2013). The calibrated age results are presented in Tables 3–5.

	Culture										
	Ser	oglazovska	ya	Jangarskaya Jangar	Orlovskaya						
Species	Kairshak III	Baibek	Tenteksor I		Varfolomeevskaya	Algay	Oroshaemoe				
Sheep – Ovis aries			1/1*/**								
Dog – Canis familiares		8/2			43/5	6/3	1/1				
Tarpan – Equus ferus			32/2	298/15	724/21	357/40	12/3				
Onager – Equus hemionus	619/12	1891/91	1290/40	615/28	714/19	266/37	28/4				
Aurochs – Bos primigenius	1/1	4/2	79/4	55/9	684/11	823/86	62/4				
Saiga – Saiga tatarica	19/3	48/7	79/9	2006/103	423/17	297/50	33/4				
Gazelle – Gazella sp.				118/24							
Red deer – Cervus elaphus	56/5	17/5	3/1	14/6	8/2	4/2					
Wild boar – Sus scrofa		5/2		2/2	18/4	3/2					
Wolf – Canis lupus	8/2	11/4	35/5		41/7						
Red fox – Vulpes vulpes	3/1	12/4		10/3	18/4						
Corsac – Vulpes corsac	2/1	35/8		57/7	13/4						
European badger – Meles meles					1/1						
Brown hare – <i>Lepus europaeus</i>	1/1	1/1		1/1		1/1					
Tolai hare – Lepus tolai		2/1									
Birds – Aves	1	1			21						
Tortoise – Chelonia					2						
Fish – Pisces		40			6	20					

Table 1 Species composition and numbers of vertebrate bone remains.

*The sheep bone has later age than other bones in the ¹⁴C dating (Table 3). **NISP/MNI (number of identified specimens/minimum number of individuals).

1600 A Vybornov et al.

	Culture								
	Ca	spian	Khvalynskaya						
Species	Kurpezhe- Molla	Oroshaemoe	Karakhuduk I	Kairshak VI	Kombak-Te				
Small stock – Ovis aries et	120/8*	22/8	152/10	168/24	94/47				
Capra hircus									
Cattle – Bos taurus			18/2	22/2	1/1				
Dog – Canis familiares				4/2					
Tarpan – Equus ferus	1/1	26/4		19/2					
Onager – Equus hemionus	78/5	9/1	17/2	21/2	25/3				
Aurochs – Bos primigenius	8/1	29/6	3/1						
Saiga – Saiga tatarica	154/5	80/17	7/2	13/2	3/1				
Red deer – Cervus elaphus	26/2			4/1	5/2				
Wild boar – Sus scrofa		2/1							
Wolf – Canis lupus	9/2								
Corsac – Vulpes corsac			10/1		3/1				

Table 2 Species composition and numbers of vertebrate bone remains, NISP/MNI.

*NISP/MNI (number of identified specimens/minimum number of individuals).

Mass spectrometry of lipids was carried out for 14 food crusts samples from pottery in the Department of Chemistry of Samara University: 1 - Kombak-Te; 2 - Tenteksor; 3 - Tenteksor I; 4 - Algay; 5 - Algay; 6 - Kairshak VI; 7 - Kairshak VI; 8 - Kairshak VI; 9 - Kairshak VI; 10 - Karakhuduk; 11 - Baibek; 12 - Baibek; 13 - Baibek; and 14 - Baibek. The deposit (m = 5 mg) from the surface of 14 ceramic samples was peeled off the scalpel and placed in glass cells, where organic compounds were derivatized for 8 hr at 60°C with a 2% solution of sulfuric acid in absolute methanol. The mixture was centrifuged for 10 min and 500 μ L of the mixture from each sample was taken. Fatty acid methyl esters adsorbed into the archeological ceramics were extracted with n-hexane (500 μ L). 400 μ L of extract was taken from the upper hexane layer and placed in Eppendorf vials. The extracts were analyzed using a gas chromatograph FK Agilent 7890A with a mass-selective detector 5975C, a gas chromatography column MP_5-M8 (length 30 m, inner diameter 0.25 mm), temperature programming from 40°C (5 min) to 100°C with a heating rate of 5°C/min, from 100°C (40 min) to 280°C with a heating rate of 10°C/min. The measurement error in the GC-MS analysis did not exceed 3%.

RESULTS AND DISCUSSION

Lipid Analysis

According to the results of lipid analysis by GC-MS, the presence of methyl esters of palmitic and stearic acids extracts from the charred crusts on ceramics were identified, as methylation of fatty acids occurs (Table 6). The predominance of these fatty acids in the sample is typical for food of animal origin. It should be noted that linolenic acid (C18: 3) and long-chain fatty acids (C20, C22, C24) are also in sufficient quantities in all samples, which may indicate the preparation of food of vegetable origin in these vessels (Table 6). One of the most useful criteria for interpreting the results of the analysis of fatty acids and understanding which products were used for cooking is the ratio of fatty acids C12: 0 / C14: 0 and C16: 0 / C18: 0. These results are presented in Table 7. Based on these criteria, it can be concluded that the food crusts of 6-9 ceramic samples includes fatty acids of vegetable origin, namely, seeds and nuts (C16: 0 / C18:

			¹⁴ C date	δ ¹³ C	Calendar age 2σ	
No.	Site, layer	Lab index*	(BP)	(‰)	(cal BC)	Material
1.	Kairshak III	Ua-41359	7775 ± 42	-28.7	6690–6490	Food crust
2.	Kairshak III	SPb-377	7700 ± 120		6830-6370	Food crust
3.	Kairshak III	Ki-14633	7190 ± 80		6230-5906	Bone
4.	Kairshak III	SPb-316	7030 ± 100		6387-6006	Bone
5.	Kairshak III	GIN-5905	6950 ± 190		6250-5500	Charcoal
6.	Baibek, object 2	Ua-50262	7937 ± 48	-29.0	7037-6684	Food crust
7.	Baibek, object 2	SPb-1712	6827 ± 100		5917-5604	Charcoal
8.	Baibek, object 1 (upper)	Poz-57060	7350 ± 50		6370–6070	Food crust
9.	Baibek, object 1 (upper)	SPb-1709	6955 ± 80		6002–5708	Animal bones
10.	Baibek, object 1 (bottom)	SPb-1714	7153 ± 90		6223–5871	Food crust
11.	Baibek, object 1 (bottom)	SPb-1721	6952 ± 80		6001-5702	Animal bones
12.	Baibek	Ua-50260	6986 ± 44	-10.0	5983-5759	Charcoal
13.	Baibek	SPb-1713	6948 ± 120		6034–5634	Charcoal
14.	Baibek	SPb-973	6955 ± 80		6002-5708	Animal bones
15.	Baibek	SPb-1710	6940 ± 80		5989-5704	Animal bones
16.	Tenteksor	Hela-3254	7261 ± 47	-28.6	6230-6030	Food crust
17.	Tenteksor	Ua-35267	6695 ± 40	-27.7	5680-5530	Charcoal from ceramics
18.	Tenteksor	SPb-315a	6540 ± 100		5640-5310	Animal bones
19.	Tenteksor	Hela-3772	4555 ± 30	-9.2	3483-3105	Sheep bones
20.	Jangar, upper layer	Hela-3255	6564 ± 44	-27.5	5575-5470	Food crust
21.	Jangar, middle layer	IGAN-2819	6870 ± 130	—	6010-5550	Charcoal

Table 3 ¹⁴C dates for sites of the northern Caspian Sea region.

*AMS radiocarbon laboratory codes: Hela – Helsinki University, Finland; Poz – Poznań Radiocarbon Laboratory Foundation of the Adam Mickiewicz University, Poland; Ua – Tandem Laboratory, University of Uppsala, Sweden; LSC radiocarbon laboratory codes: GIN – Geological Institute, Russian Academy of Sciences, Moscow, Russia; IGAN – Institute of Geography, Russian Academy of Sciences, Moscow, Russia; Ki – Institute of Radio- Geochemistry of the Environment, Ukraine; SPb – Herzen State University, St. Petersburg, Russia.

0 = 0-18, C12: 0 / C14: 0 > 0.15) (Eerkens 2005). Food of animal origin was prepared in ceramic vessels of 1-4 and 10-14 (acid ratios C16: 0 / C18: 0 < 7, C12: 0 / C14: 0 < 0.15). Thus, it can be argued with a high degree of probability that food of animal and vegetable origin was prepared in these vessels. Small amounts of residues of short-chain fatty acids (caproic C6: 0, caprylic C8: 0 and capric C10: 0) are found in samples 7, 8, and 9 (Table 6). These vessels could have been used to prepare dairy products (Evershed 2002), but this may also be related to the decomposition of longer chain acids.

Archaeozoology and Radiocarbon Dating - Caspian Sea Region

Kairshak Type of Sites (Earlier Stage of the Neolithic Seroglazovskaya Culture)

In the southern part of the Lower Volga basin (the semiarid zone of the Northern Caspian Sea region), the Kairshak III site was investigated, which is the *locus classicus* of the Kairshak

Table 4 ¹⁴ C dates for sites of the Low Volga region.
--

No.	Site, layer	Lab index*	¹⁴ C date (BP)	δ ¹³ C (‰)	Calendar age 2 σ (cal BC)	Material
1.	Varfolomeevskaya, layer 3	GIN 6546	6980 ± 200		6246–5519	Charcoal
2.	Algay, bottom layer	SPb-2144	7284 ± 80		6359–6008	Humic acids
3.	Oroshaemoe, bottom layer	SPb-2141	7245 ± 60		6227-6015	Charcoal
4.	Varfolomeevskaya, layer 2B	SPb-941	7100 ± 110		6211-5753	Food crusts
5.	Varfolomeevskaya, layer 2B	Ua 41360	7034 ± 41	-28.0	6008-5837	Food crusts
6.	Oroshaemoe, bottom layer	SPb-2090	6889 ± 100		5933-5631	Animal bones
7.	Varfolomeevskaya, layer 2A	Ua-41362	6693 ± 39	-28.3	5700-5533	Food crusts
8.	Varfolomeevskaya, layer 2A	SPb-938	6650 ± 150		5872-5317	Food crusts
9.	Varfolomeevskaya, layer 2A	Ua-41361	6544 ± 38	-25.1	5613-5392	Food crusts
10.	Varfolomeevskaya, layer 2A	SPb-937	6363 ± 150		5617-4994	Food crusts
11.	Algay, bottom layer	Poz-65198	6800 ± 40		5741-5631	Food crusts
12.	Algay, bottom layer	SPb-1510	6820 ± 80		5889-5614	Animal bones
13.	Algay, bottom layer	SPb-1509	6654 ± 80		5708-5479	Animal bones
14.	Algay, bottom layer	SPb-1478	6577 ± 80		5641-5374	Animal bones
15.	Algay, bottom layer	SPb-1411	6360 ± 250		5742-4723	Charcoal
16.	Algay, bottom layer	SPb-1477	6479 ± 70		5560-5316	Animal bones
17.	Algay, bottom layer	Poz-76004	6490 ± 40		5527-5367	Charcoal
18.	Algay, bottom layer	AAR 21892	6318 ± 33		5361-5221	Animal bones
19.	Algay, bottom layer	SPb-2038	6284 ± 100		5472-5018	Animal bones
20.	Algay, bottom layer	AAR 21891	6245 ± 32		5309-5076	Food crusts
21.	Algay, bottom layer	AAR 21894	7580 ± 46		6561–6368	Fish bone
22.	Algay, bottom layer	AAR 21893	6605 ± 32		5617-5487	Charcoal

*AMS radiocarbon laboratory codes: AAR – the University of Aarhus, Denmark; Poz – Poznań Radiocarbon Laboratory Foundation of the Adam Mickiewicz University, Poland; Ua – Tandem Laboratory, University of Uppsala, Sweden; LSC radiocarbon laboratory codes: GIN – Geological Institute, Russian Academy of Sciences, Moscow, Russia; SPb – Herzen State University, St. Petersburg, Russia.

No.	Site, layer	Lab index*	¹⁴ C date (BP)	δ ¹³ C (‰)	Calendar age 2σ (cal BC)	Material
1.	Kombakte	GIN-6226	6000 ± 150		5290-4550	Charcoal
2.	Oroshaemoe, middle layer	SPb-2091	5934 ± 100		5060-4547	Animal bone
3.	Algay, upper layer	SPb-196	5875 ± 60		4856-4580	Animal bone
4.	Oroshaemoe, middle layer	UGAMS-23059	5806 ± 26		4724–4557	Domestic sheep bone
5.	Algay, upper layer	SPb-1475	5720 ± 120		4837–4342	Animal bone
6.	Oroshaemoe, upper layer	SPb-1474	5667 ± 100		4725–4336	Animal bone
7.	Kairshak VI	AAR-26175	5664 ± 80	_	4687–4356	Food crusts
8.	Kombakte	AAR-22804	5626 ± 51		4550-4350	food crusts
9.	Khvalinian burial	GrA-24442	5840 ± 40	-20.77	4779–4683	Human bones
10.	Khvalinian burial	GrA-29178	5565 ± 40	-17.86	4447-4417	Sheep bones

Table 5 14 C dates for sites of the Cis-Caspian (1–5) and Khvalinian cultures (6–10).

*AMS radiocarbon laboratory codes: AAR – the University of Aarhus, Denmark; GrA – Groningen, Netherlands; UGAMS – the University of Georgia, USA; LSC radiocarbon laboratory codes: GIN – Geological Institute, Russian Academy of Sciences, Moscow, Russia; SPb – Herzen State University, St. Petersburg, Russia.

archaeological type. The Kairshak type of sites was defined based on ceramic typology and the slab stone tools with geometric microliths in the shape of segments (Vasiliev et al. 1989). Organic silt (probably from the river floodplain) with admixture of shells was the basic raw material used in pottery making. The pottery is flat-bottomed, with pinned and incised ornamentation. On the base of typology, researchers (Koltsov 2004; Yudin 2004; Vybornov 2008) described these materials as the Earliest stage of the Neolithic Seroglazovskaya culture. The Neolithic Seroglazovskaya culture was defined by Meleniev taking into consideration the sites with complex of pottery with pinned and incised ornamentation and stone tools with geometric microlites (Melentiev 1975). The sites with homogenic complexes were not known in this time. Preliminary results about diet on the sites of Kairshak type have been considered earlier elsewhere (Barynkin and Kozin 1998). However, to be useful, they should be derived from statistically significant sites containing preserved animal bones. Based on typology, the researchers (Koltsov 2004; Yudin 2004; Vybornov 2008) describe these materials as the Earliest stage of the Neolithic Seroglazovskaya culture. The authors were not able to determine the economy of the site.

During 2013–2017, the Baibek site of Kairshak type was excavated (Grechkina et al. 2014). This is a single layered site and the artifacts occurred in houses and household pits. Animal bones were found inside these structures in a clear archaeological context. Onager, saiga antelope, red deer, aurochs, wild-boar, corsac fox, wolf, and birds (Table 1) were identified at Kairshak type sites of the Seroglazovskaya culture (both Baibek and Kairshak-III) in the North Caspian Sea region (Vybornov 2008; Grechkina et al. 2014). The dog (*Canis familiaris*) is the only example of domestic animals. While Kairshak III is located far from water bodies, Baibek is situated near a river and a lot of fish bones (stellate sturgeon, bream, starlet, catfish, pike perch, perch, pike) were found at this site. This is evidence of consumption of freshwater resources by people, indicating a diverse economy and diet in the southern part of the Low Volga basin.

						(Concentr	ation (%)					
	Sample													
Fatty acid	1	2	3	4	5	6	7	8	9	10	11	12	13	14
C 6:0 caproic							0.6	2.1	2.6	_				
C 8:0 caprylic		_			_		1.3	2.7	3.7			_		
C 10:0 capric							0.8	2.5						
C 12:0 lauric		_				0.3	0.6	2.2	3.5					
C 14:0 myristic		_	18.6			0.5	0.2	0.9	0.7	0.6				0.8
C 14:1 myristoleic			18.6										0.9	
C 16:0 palmitic	34.6	3.5	11.4		0.6	0.8	0.6	3.3	2.1	1.4	13.5	11.5	3.5	7.2
C 16:1 palmitoleic				0.9	—			—				—		
C 18:0 stearic	54.3	2.3	14.3	0.4	—	1.6	0.8	7.5	3.7	2.2	5.9	—	1.0	0.9
C 18:1 oleic		0.8	_	1.6	1.5	2.3	0.9	0.6	1.0		13.4	4.6	3.0	4.8
C 18:2 linoleic	11.0	_	4.8	0.7	0.7	1.3	0.5	_	0.9	0.5	18.6	4.3	_	1.4
C 18:3 linolenic		29.8	_	33.8	33.3	30.7	22.8	39.7	31.2	23.6	26.0	69.1	32.8	57.1
C 20:0 arachidi		1.3	_	_	2.3	2.6	4	1.9	1.2	1.3	6.5	5.7	_	5.1
C 20:1 gondoic		_	21.0	_	_	_	_	_				_	_	
C 20:2 eicosadienoic		1.6	11.3	4.0	_	_	_	15.0	19.4	20.6		_	_	
C 22:0 behenic					7.6	4.7		—			16.0	4.7	1.3	3.1
C 22:1 erucic		10.6		13.8	12.6	15.0	13.0	—		1.5		—		19.4
C 22:2 docosadienoic		45.5		40.3	36.3	36.0	28.5		4.6	5.0				
C 24:0 lignoceric		4.5		4.6	5.1	4.0	15.5	11.0	16.0	17.4			49.7	
C 24:1 selacholeic		_			_		0.8	3.6	3.5	6.4		_	7.7	_

 Table 6
 Concentrations of fatty acids found in the deposit from the walls of 14 samples of ceramics.

		Concentr	ation (%)			
Sample	C12:0	C14:0	C16:0	C18:0	C12:0/C14:0	C16:0/C18:0
1.		_	34.6	54.3		0.64
2.			3.5	2.3		1.48
3.		18.6	11.4	14.3	0	0.80
4.				0.4		0
5.			0.6			
6.	0.3	0.5	0.8	1.6	0.54	0.53
7.	0.6	0.2	0.6	0.8	2.73	0.75
8.	2.2	0.9	3.3	7.5	2.37	0.44
9	3.5	0.7	2.1	3.7	5.30	0.57
10.		0.6	1.4	2.2	0	0.64
11.			13.5	5.9	_	2.30
12.			11.5		_	
13.			3.5	1.0		3.45
14.		0.8	7.2	0.9	0	7.68

Table 7 Concentrations and ratios of fatty acids C12:0/C14:0 and C16:0/C18:0, allowing interpretation of the results.*

*C12:0 – lauric acid; C14:0 – myristic acid; C16:0 – palmitic acid; C18:0 – stearic acid.

Some of the ¹⁴C results on the Kairshak sites were controversial (Vybornov 2008; Grechkina et al. 2014). Over several years, about 50 ¹⁴C dates were obtained for these sites. Most of these dates were obtained on organics from pottery and food crusts and they fall into the interval from ca. 7000 to 6400 cal BC (Vybornov 2008). The dates on animal bones and charcoal fall into the interval ca. 6300-5500 cal BC (Table 3: 4-6). Two dates on food crusts (Table 3: 1-2) from Kairshak III appear to be outliers. Besides the 14 C dates on food crusts (ca. 6800–6700 cal BC) from this site there are the ¹⁴C dates on bones (Table 3: 3–4) and on charcoal (Table 3: 5), which are in interval of 6300–6200 cal BC. Probably, the dates on food crusts are older because of the reservoir effect (a δ^{13} C value of -28.7% indicates the presence of freshwater resources). From Baibek, we obtained two dates on food crusts and charcoal from the same construction No. 2 (Table 3: 7-8). These dates differ by about 1000 years. We suggest the reservoir effect influenced the older food crust ($\delta^{13}C = -29.0\%$). Similar results were recorded on the dates from other house pits. The difference between ¹⁴C dates on food crusts and animal bones is 200-300 yr (Table 3: 9–12). The most valid ¹⁴C date for Baibek is about 6000 cal BC, supported by 14 C dates on charcoal and animal bones (Table 3: 13–16). So, the interval of development of the Kairshak type of sites is from ca. 6300 to 5700 cal BC.

Tenteksor Type of Sites (the Later Stage of the Neolithic Seroglazovskaya Culture)

On the base of typological analysis, researchers (Koltsov 2004; Yudin 2004; Vybornov 2008) considered the materials of the Tenteksor site as the Later stage of the Neolithic Seroglazovskaya culture. The material culture is presented by pottery with retreating pinned ornamentation with complex geometric design and stone tools with trapezoidal microliths. The Jangar site is located on the right shore of the Volga in the northwestern part of the Caspian Sea and, according to some investigations (Koltsov 2004) has been attributed to the Jangar culture.

At later sites of the Tenteksor type in this region, onager, saiga antelope, red deer, aurochs, tarpan, and wolf were identified (Table 1). At the Tenteksor site as well as on other sites of the

Seroglazovskaya culture, bones of onager are predominant, exceeding 80% of bone material. Some archaeologists (Vasiliev et al. 1986) considered the possibility of the presence of domestic sheep on this site. One sheep bone was found in the deposits of Tenteksor. The AMS date of this bone (Table 3: 20) deviates from other ¹⁴C dates on different organic materials for the Tenteksor site (Table 3: 18–19). However, the hypothesis of the presence of domestic sheep at Tenteksor cannot been confirmed on the basis of this limited data.

The chronology of Tenteksor type of sites was poorly developed. On the base of the first obtained date of 5500 ± 150 BP (4684–4036 cal BC), some archaeologists (Koltsov 2004) suggested the coexistence of the Tenteksor culture and the Khvalinian Eneolithic culture. A series of ¹⁴C dates for several sites of the Tenteksor type was obtained recently (Vybornov 2008). There are discrepancies in these dates. One of the dates was obtained on food crusts from pottery (Table 3: 17). Stable isotope analysis showed a value of $\delta^{13}C = -28.6\%$. Probably, in this case the reservoir effect influenced value of this date also: This date (7261 ± 47 BP, Hela 3254) is the same age as the date on shells (7235 ± 45 BP [Ua_35226]; Zaitseva et al. 2009). Charcoal from this ceramic fragment is 500 years younger than the date on shells (Table 3: 18). The last date was supported also by the date on animal bones from this site (Table 3: 19). Consequently, we propose that sites of the Tenteksor type date to 5700 to 5400 cal BC.

The Jangar Site

The Jangar site is located on the right shore of the Volga in the northwestern part of the Caspian Sea region (Koltsov 2004). The main feature of the Jangar site is its location. This site is located in the Northern part of the steppe zone, whereas the Kairshak site is located in the semi-desert zone. Thus, the fauna at these sites are expected to be different. Unlike the sites of Kairshak type the dominant species at the Jangar site are saiga (Table 1). Gazelle bones occurred as well, but they have not been found in the sites of the northern Caspian Sea region. On the other hand, the bones of tarpan were found in the Tenteksor site. Saiga and gazelle might occur at Jangar as it lies farther north to the steppe zone than Kairshak. Aurochs, red deer, corsac fox, wild boar, and wolf bones were determined in the layers of Jangar site. The suggestion about horse domestication (Gasilin et al. 2008) was not confirmed by palaeozoological investigations. Thereby the meat diet of the Jangar culture has some distinctions from habitants of the northern Caspian Sea region.

The chronology of this cultural complex was based on only two dates (6100 ± 70 BP [Le-2564] for the second cultural layer and 5890 ± 70 BP [Le-2901] for the first cultural layer) that were obtained on charcoal in the 1980s. Based on these dates, the Jangar complex would coexist with the later Cis-Caspian culture. In the last years, new dates on different organic materials were obtained. The ¹⁴C date on food crusts (Table 3: 21) from pottery of the second cultural layer is 6564 ± 44 BP (6010-5550 cal BC) (HeLa-3255) and the ¹⁴C date on charcoal from the first cultural layer is 6870 ± 130 BP (5575-5470 cal BC) (IGAN-2819) (Table 3: 21–22). The most reliable date of Jangar is thus from ca. 6000 to 5500 cal BC.

Archaeozoology and Radiocarbon Dating – Steppe Povolzhie Region

Sites of the Orlovskaya Neolithic Culture

In the steppe zone of the Low Volga region, just one site, Varfolomeevskaya, was investigated by the zooarchaeological method (Yudin 2004; Gasilin et al. 2008). In 2014–2017, assemblages of the Orlovskaya culture from the site Algay and the lower layer of Oroshaemoe I were studied (Vybornov et al. 2015, 2016a, 2016b, 2017). It needs to be noted that these three sites have good

stratigraphy. According to stratigraphy three cultural layers were distinguished: the Early Neolithic, the Middle Neolithic, and the Eneolithic. These periods were confirmed by ^{14}C dates also. The results of paleozoological analysis showed that unlike Kairshak and Jangar, the bones of aurochs and other ungulates prevailed in the cultural layers of the Orlovskaya culture (Table 1). Bones of saiga, tarpan, and onager were found. Bones of red deer, wild boar, wolf, fox, hare, and fish were less numerous. Bird bones were rare. The presence of domestic sheep bones in the cultural layer of the Orlovskaya culture was not supported (Gasilin et al. 2008). The dog (*Canis familiaris*) is the only domestic animal. There are noted some differences in the meat diet between the inhabitants of the steppe Povolzhie region and the Caspian Sea region.

The chronological framework of the Orlovskaya culture had been based on seven ¹⁴C dates for four layers of Varfolomeevskaya. Some dates are disputable. At present, for the Orlovskaya culture 50 ¹⁴C dates on bones, charcoal, food crusts and organics from pottery were obtained (Vybornov 2008; Vybornov et al. 2012, 2016c). The oldest phase of the Orlovskaya culture is dated to ca. 6200 cal BC (Table 4: 1–4). The next stage is dated to 6000–5620 cal BC (Table 4: 5–6, 11–12). Interestingly, the ¹⁴C dates on animal bones and food crusts agree, indicating only a minimal reservoir effect. The dates on charcoal and fish bones from the same cultural layer of Algay differ by 1000 years (Table 4: 21–22). The sequence of obtained dates, with considering outliers, is supported also by the clear stratigraphic layer successions of the Algay, Oroshaemoe, and Varfolomeevskaya sites. The final phase of the Orlovskaya culture is dated to ca. 5600–5300 cal BC (Table 4: 9, 10, 13–20).

Sites of the Cis-Caspian Culture

In the Low Volga basin, apart from the sites of Kairshak, Tenteksor, Jangar, and Orlovsky types, another cultural group of sites was distinguished. This is the Cis-Caspian culture (Melentiev 1976; Vasiliev 1981; Yudin 2012). Prior to 2007, only a few sites with artifacts of the Cis-Caspian culture had been excavated. During 2014–2017, the new site Oroshaemoe I of the Cis-Caspian culture was excavated (Vybornov et al. 2015, 2017). The well-preserved cultural layer includes numerous animal bones. Species such as saiga, aurochs, tarpan, onager, red dear, wild boar, and wolf were identified (Table 2). This selection is similar to the bone assemblages of Orlovskaya and Tenteksor types. But there are some differences. In the layer of the Cis-Caspian culture, the bones of domestic sheep and goat were identified (Table 2). The bones of domestic sheep and goat were also found in the Kurpezhe-Molla site, which is located in the northern Caspian Sea region (Table 2). Before this study, the first domestic ungulates were assigned to the Khvalinian Eneolithic culture (Kuzmina 1988). The latest results agree with the conclusion that domestic animals appeared together with carriers of the Cis-Caspian culture (Table 2, Table 4: 5).

The chronology of the Cis-Caspian culture was defined on the basis of a series of dates obtained on different type of organics from different archaeological sites (Table 5: 1–5). One of the dates was obtained on bones of domestic sheep (Table 5: 4). It is a temporal marker for the earliest appearance of domestic sheep in the Cis-Caspian culture. Based on the new results, we can date the Cis-Caspian culture to the interval from ca. 5200 to 4700 cal BC.

The results of the lipid analysis showed that animal or plant food had been cooked inside of the pottery from Kairshak III, Baibek, Tenteksor, Algay and Varfolomeevka sites. Fatty acids of fish have not been identified in those vessels.

Eneolithic Sites

The sites of the Khvalinian culture were spread over the territory of the Low Volga basin. The first copper artifacts were found in the assemblages of the Khvalinian culture (Vasiliev 1981; Yudin 2012). In the single layered sites of this culture (Kombakte and Kairshak VI), animal bones were sampled. The bones of onager, saiga, tarpan, red deer, aurochs, and corsac fox were identified. The harpoons found in burials are evidence of fishing (Barynkin 1989; Agapov et al. 1990). Domestic animals are represented by sheep and cattle (Table 2). The meat component of the diet of the Khvalinian culture was thus more diverse than in the Neolithic period.

Twenty-five ¹⁴C dates are available for the Khvalinian culture, spanning from ca. 5100 to 4700 cal BC (Morgunova 2011). This interval coincides with the Cis-Caspian culture. However, most of these dates were obtained on human bones from the Khvalinian burials and they could be older because of the reservoir effect. There are differences between dates on human bones (Table 5: 9) and animal bones (Table 5: 10) from the same burial of about 300 years (Shishlina et al. 2006). Two samples of organics from pottery are dated to ca. 4300 cal BC. There are several dates on different organics (Table 5: 6–8) in the interval 4700–4400 cal BC. These data show sequential development of the Cis-Caspian and Khvalinian cultures.

In the Eneolithic sites of Karakhuduk and Kombakte, an animal diet prevailed. At the Kairshak VI site, vegetarian food prevailed. It is very important to note the presence of short-chain fatty acids in the food crusts from the pottery of Kairshak VI site, as they are a characteristic of dairy products.

CONCLUSION

These results represent the first data about the diet of people of different cultures in the Low Volga basin. Wild species prevailed on the Kairshak and Tenteksor type of sites of the Seroglazovskaya culture of the northern Caspian Sea region, at the sites of the Jungar culture located in the northwestern Caspian Sea region and at the sites of the Orlovskaya Neolithic culture of the Low Volga region. In the materials of the Eneolithic cultures (Cis-Caspian and Khvalinian), domestic animal bones (sheep, cattle) were identified. The diet included both wild and domestic animals. The large series of ^{14}C dates gave the possibility to establish a detailed chronology for these cultures. For the first time in the study of the Neolithic and Eneolithic of Russia, lipid analysis of charred food crusts by mass spectrometry was carried out for diet reconstruction. Further investigations of lipids and stable isotopes in food crusts are needed for a detailed analysis of food preparation and to specify the chronological frameworks of those cultures. It is necessary to carry out additional studies of the samples of food crusts as well as the isotopic analysis of the C16: 0 and C18: 0 acids (Dudd and Evershed 1998). This large series of ¹⁴C dates obtained on different organic materials (bone, food crusts, charcoal) is very important because it allowed to us reconstruct the diet during the Neolithic-Eneolithic periods in the Lower Volga basin. The first Neolithic sites in Eastern Europe developed exactly in this region. As a result, the chronological stages of the Neolithic and the time of transition to the Eneolithic on this territory were determined. According to the dates obtained, we established the time of cattle herding appearance in Eastern Europe. It is worth noting that in Europe, the development of cattle herding has earlier dates (see Anthony 2007).

ACKNOWLEDGMENTS

These investigations were supported by the project 33.1907.2017 state order of Russian Ministry of Education and Science. The study is partially supported by program of UD RAS, project No. 18-4-4-3.

REFERENCES

- Agapov SA, Vasiliev IB, Pestrikova VI. 1990. *Khvalynsky eneolitichesky mogilnik [Khvalinian Neolithic burial]*. Saratov: Saratov University Publishing House. 160 p. In Russian.
- Anthony DW. 2007. The Horse, The Wheel, and Language: How Bronze-Age Riders From the Eurasian Steppes Shaped the Modern World. Princeton (NJ): Princeton University Press. 553 p.
- Arslanov KA, Svezhentsev YS. 1993. An improved method for radiocarbon dating fossil bones. *Radiocarbon* 35(3):387–91.
- Barynkin PP. 1989. Eneoliticheskiy pamyatnik Kair-Shak VI iz yuzhnoy chasti Volgo-Uralskogo mezhdurechiya [Eneolithic site of Kairshak VI from Southern part of Volga-Ural basin] In: Merpert N, editor. *Neolit i eneolit Severnogo Prikaspiya*. Kuibyshev: Kuibyshev State Pedagogical University, p 106–18. In Russian.
- Barynkin PP, Kozin EV. 1998. Prirodnoklimaticheskie i kulturno-demograficheskie prozessi v Severnom Prikaspii v rannem i srednem golozene [Environmental-climatic and culturaldemographic processes in the Northern Caspian Sea region in Early and Middle Holocene]. The Problems of ancient history of Northern Caspian Sea region. Materials of scientific articles. Samara Pedagogical University Publishing House. p 66–82. In Russian.
- Bronk Ramsey C, Scott M, van der Plicht H. 2013. Calibration for archaeological and environmental terrestrial samples in the time range 26–50 ka cal BP. *Radiocarbon* 55(4):2021–7.
- Cottereau E, Arnold M, Moreau C, Baqué D, Bavay D, Caffy I, Comby C, Dumoulin J-P, Hain S, Perron M, Salomon J, Setti V. 2007. Artemis, the new 14C AMS at LMC14 in Saclay, France. *Radiocarbon* 49(2):291–9.
- Dudd SN, Evershed RP. 1998. Direct demonstration of milk as an element of archaeological economies. Science 282(5393):1478–81.
- Eerkens JW. 2005. GC-MS analysis and fatty acids rations of archeological potsherds from the western Great Basin of North America. *Archaeometry* 47(I.1):83–102.
- Evershed RP, Dudd SN, Copley MS, Berstan R, Stott AW, Mottram H, Buckley SA, Crossman Z. 2002. Chemistry of archaeological animal fats. *Account* of Chemical Research 35(8):660–8.
- Gasilin VV, Kosintsev PA, Sablin MV. 2008. Fauna neoliticheskoy stoyanki Varfolomeevskaya v stepnom Povolzhe [The fauna of Neolithic site of Varfolomeevskaya in steppe Povolzhie. In: Kosintsev P, editor. *Fauna i flora severnoy Evrazii* v pozdnem kaynozoe. Ekaterinburg, Chelyabinsk. p 25–100. In Russian.
- Grechkina TY, Vybornov AA, Kutukov DV. 2014. Novaya ranneneoliticheskaya stoyanka Baybek v Severnom Prikaspii [New Early Neolithic site of

Baibek in the Northern Cis-Caspian]. *Samarskiy nauchnyy vestnik* 3:79–90. In Russian.

- Koltsov PM. 2004. *Poseleniye Jangar*. Moscow: Novyi Hronograf: 156 p. In Russian.
- Kuzmina IE. 1988. Mlekopitayushchie Severnogo Prikaspiya [Mammals of Northern Cis-Caspian]. In: Merpert N, editor. Arheologicheskie kultury Severnogo Prikaspiya. Kuibyshev: Kuibyshev State Pedagogical University. p 178–88. In Russian.
- Melentiev AN. 1975. Pamyatniki Seroglazovskaya culture (Neolit Severnogo Prikaspiya) [The sites of the Seroglasov culture (the Neolithic of the Northern Caspian Sea region)]. Short Reports of the Institute of Archaeology 141:112–7. In Russian.
- Melentiev AN. 1976. Pamjatniki neolita Severnogo Prikaspija (pamjatniki prikaspiyskogo tipa) [The sites of the Neolithic of the Northern Caspian Sea region)]. In: Merpert N, editor. Problemy archeologii Povolzhya i Priuralya Kuibyshev. Kuibyshev State Pedagogical University. p 13–4. In Russian.
- Morgunova NL. 2011. Eneolit Volzhsko-Uralskogo mezhdurechja [Eneolithic of Volga-Ural basin]. Orenburg: Orenburg State Pedagogical University. 220 p. In Russian.
- Shishlina NI, Plicht J, Zazovskaya EP, Sevastyanov VS, Chichagova OA. 2006. K voprosu o radiouglerodnom vozraste eneoliticeskih kultur Evraziskoy stepi [To the question of radiocarbon age of Eneolithic cultures of Evrasian steppe]. Voprosy archeologii Povolzhya 4:134–40. In Russian.
- Yudin AI. 2004. Varfolomeevskaya stoyanka i neolit stepnogo Povolzhya [The Varfolomeevskaya site in the Neolithic of steppe Povolzhie]. Saratov: Saratov University Press. 200 p. In Russian.
- Yudin AI. 2012. Poselenie Kumyska I eneolita stepnogo Povolzhya [Kumyska I settlement of Eneolithic of steppe Povolzhie]. Saratov: Nauchnaya kniga. 212 p. In Russian.
- Vasiliev IB. 1981. Eneolit Povolzhya [Eneolithic of Povolzhie]. Kuibyshev: Kuibyshev State Pedagogical University: 80 p. In Russian.
- Vasiliev IB, Vybornov AA, Kozin EV. 1986 Pozdneneoliticheskaya stojanka Tenteksor v Severnom Prikaspii [The late Neolithic site of Tenteksor in the Northern Caspian Sea]. In: Merpert N, editor. *Drevnii kultiry Severnogo Prikaspija*. Kuibyshev: Kuibyshev State Pedagogical University. p 6–31. In Russian.
- Vasiliev IB, Vybornov AA, Kozin EV. 1989. Issledovanie neoliticheskoy stojanki Kairshak III [The investigations of Neolithic site of Kairshak III].
 In: Merpert N, editor. *Neolit i eneolit Severnogo Prikaspiya*. Kuibyshev: Kuibyshev State Pedagogical University. p 18–45. In Russian.
- Vybornov AA. 2008. Neolit Volgo-Kamya [Neolithic of Volgo-Kama]. Samara: Samara State University of Social Sciences and Education. 490 p. In Russian.

- Vybornov A, Zaitseva G, Kovaliukh N, Kulkova M, Skripkin V, Possnert G. 2012. Chronological problems with neolithization of the northern Caspian sea area and the forest-steppe Povolzhye region. *Radiocarbon* 54(3–4):795–9.
- Vybornov AA, Yudin AI, Vasileva IN, Kosintsev PA, Kulkova MA, Goslar T, Doga NS. 2015. Novye materialy neolita Nizhnego Povolzhya [New materials of Neolithic of the Low Volga region]. *Izvestiya Samarskogo nauchnogo tsentra Rossiyskoy akademii nauk* 17(3):235–41. In Russian.
- Vybornov AA, Yudin AI, Vasileva IN, Kosintsev PA, Kulkova MA, Goslar T, Philippsen B, Baratskov A. 2016a. Neoliticheskaya stoyanka Algay v Nizhnem Povolzhje [The Neolithic site Algay in the Low Volga basin]. Samarskiy nauchny vestnik 3(16):61–8. In Russian.
- Vybornov AA, Yudin AI, Vasileva IN, Kosintsev PA, Kulkova MA, Goslar T, Doga NS, Popov AS. 2016b. Issledovanie poseleniya Oroshaemoe v Nizhnem Povolzhje [The researches of Oroshaemoe site in the Low Volga basin]. Izvestiya

Samarskogo nauchnogo tsentra Rossiyskoy akademii nauk 18(3):140–5. In Russian.

- Vybornov AA, Yudin AI, Kulkova MA, Goslar T, Possntrt G, Philippsen B. 2016c. Radiouglerodnye danny dlja hronologii neolita Nizhnego Povolzhja [The radiocarbon data for chronology of Neolithic of the Low Volga basin]. In: Mazurkevich A, Dolbunova E, Kulkova M, editors. *Radioglerodnaja hronologija epohi neolita Vostochnoy Evropy* VII–III tys. do n.e. Smolensk: Svitok. p 59–70. In Russian.
- Vybornov AA, Yudin AI, Vasileva IN, Kosintsev PA, Kulkova MA, Doga NS, Popov AS. 2017. Novye materialy issledovaniy na poselenii Oroshaemoe v Nizhnem Povolzhje [New materials of investigation of Oroshaemoe site in the Low Volaga basin]. *Izvestiya Samarskogo nauchnogo tsentra Rossiyskoy akademii nauk* 19(3):185–90. In Russian.
- Zaitseva G, Skripkin V, Kovaliukh N, Possnert G, Doluhanov P, Vybornov A. 2009. Radiocarbon dating of Neolithic pottery. *Radiocarbon* 51 (2):795–801.