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MODERN FRESHWATER RESERVOIR OFFSETS IN THE EURASIAN STEPPE: IMPLICATIONS FOR ARCHAEOLOGY

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ABSTRACT. This paper presents the results of the first broad-scale study of modern freshwater reservoir effects (FREs) in various regions of the Eurasian Steppe, associated with archaeological sites. The aim of this work was not only to demonstrate the widespread variability of modern FREs in the region, but also to draw the attention of specialists working in the area to the necessity of taking into account this important and still not fully understood factor involving radiocarbon dating of human and some faunal remains from archaeological sites. To identify modern FREs, modern fish of different species from 10 regions of Siberia and Kazakhstan have been subjected to accelerator mass spectrometry radiocarbon (AMS ^{14}C) dating and stable carbon and nitrogen isotope analysis, and the results are compared with the existing data from previous research. Freshwater reservoir offsets have been detected in all analyzed regions, with the exception of Kharga Lake (Buryatia, Russia) and Kyzylkoi River (central Kazakhstan), varying not only between, but also within regions depending on fish species. The most significant offset in this study has been recorded for the Chuya River basin (Altai Mountains, 1097 ± 40 ^{14}C yr), though not as high as observed in previous research for the Caspian lowlands (1477 ± 52 and 1037 ± 52 ^{14}C yr) and Upper Lena River basin (Lake Baikal area, 1981 ± 30 ^{14}C yr). Both δ^{13}C and δ^{15}N values have been measured with the majority of samples reflecting C_3 ecology of local reservoirs and δ^{15}N depending on the diet of particular species, with predatory species such as pike, perch, and burbot demonstrating the highest δ^{15}N. No general relationship has been observed between freshwater reservoir offsets and either δ^{13}C or δ^{15}N values of the samples.

KEYWORDS: freshwater reservoir effects, radiocarbon dating, Eurasian Steppe, modern fish.

INTRODUCTION

In archaeology, radiocarbon (^{14}C) dating is one of the most accurate and commonly used methods for both determining the age of individual organic samples and building reliable chronologies for archaeological cultures and historical events. Reservoir effects may become a factor introducing error into individual ^{14}C dates and entire chronological reconstructions. Specialists often sample human or faunal bones for ^{14}C dating because these are frequently of particular interest given the research questions; however, if a part of their diet comes from a reservoir with lower ^{14}C than the atmospheric level (such as oceanic or in some cases inland fresh water), the sample may be affected by a reservoir offset (for a detailed description of the chemistry and causes of reservoir effects see Deevey et al. 1954; Deevey and Stuiver 1964; Keaveney and Reimer 2012; Wood et al. 2013; Fernandes et al. 2016 and others).

As has been pointed out previously (e.g. Svyatko et al. 2015, 2016), research on the extent of the freshwater reservoir effect (FRE) is rather scarce compared to the studies of marine reservoir effects, although such studies are rapidly increasing in number. Published FRE (modern and archaeological) studies in Siberia and the Eurasian Steppe have focused on the north Peri-Caspian Sea region and the lower course of the Don River (Shishlina et al. 2007, 2009, 2010, 2012, 2014; Motuzaite-Matuzeviciute et al. 2015; van der Plicht et al. 2016), the middle and lower Dnieper basin (Lillie et al. 2009), the north Caucasus (Higham et al. 2010), the Upper Lena River and Baikal Lake regions (Nomokonova et al. 2013; Schulting et al. 2014, 2015), northeast Kazakhstan (Svyatko et al. 2015), the Serteyka River in the Smolensk Oblast (Kulkova et al. 2015), the site of Minino on the shores of Kubenskoye Lake in Vologda Oblast (Wood et al. 2013), and the Minusinsk basin of southern Siberia (Svyatko et al. 2016). Several studies have also assessed the extent of the reservoir offsets within the Caspian (Olsson 1980; Arslanov and Tertychnaya 1983;

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Karpytchev 1993; Kuzmin et al. 2007; Leroy et al. 2007) and Aral Seas (Kuzmin et al. 2007), both technically being lakes because they not connected to the ocean.

The aim of this study is to further access the variation in modern FREs across various regions of the Eurasian Steppe, especially from water sources associated with archaeological sites, through ^{14}C dating of modern fish samples, and combining the results with the existing data from previous research. As such, the study is particularly topical for archaeologists working in the inland areas of the Eurasian Steppe, providing an indication of the extent of possible ^{14}C offsets. The results will allow us to re-evaluate the potential and limitations of the ^{14}C dating of bone material, most notably human remains, as well as samples containing fish and/or shell remains (e.g. pottery made with inclusions of fish bones and shells, etc.) or food crusts on pottery.

Variability and Sources of FRE

The "aging" of a carbon compound starts when it ceases to exchange with its surroundings, e.g. with the death of a living organism. ^{14}C has a half-life of 5730 yr, so after ca. 50,000 yr no detectable ^{14}C remains. As many sedimentary rocks are composed of the skeletal fragments of marine organisms that died millions of years ago, they represent a major depository of ^{14}C-free or "old" carbon. Therefore, a major source of "old" carbon in freshwater flowing over limestone is dissolved inorganic carbon from weathering of ^{14}C-free carbonate minerals (e.g. Sveinbjornsdottir et al. 1995), together with inputs of old soil humus (Keith and Anderson 1963) or decaying organic matter that is washed into the water from the catchment (Goh 1991). A long residence time of water in a lake or aquifer (leading to a slow CO_2 exchange between the atmosphere and the water) can also lead to an older ^{14}C age of the water (Broecker and Walton 1959; Hakansson 1976; Geyh et al. 1998; Culleton 2006). Other contributing factors include melting glaciers and the consequent release of "old" carbon dioxide (CO_2) (Hall and Henderson 2001; Osipov and Khlystov 2010), underwater output of methane hydrates (Prokopenko and Williams 2004) or methane from chalk beds (Trimmer et al. 2009), and geothermal activity (Ascough et al. 2010; Higham et al. 2010).

The extent of the FRE in a region is thought to be closely related to the geological composition of the underlying bedrock or surficial deposits. Keaveney and Reimer (2012) demonstrated a high correlation between the FRE and carbonate alkalinity in modern lakes in Britain and Ireland. One can expect a greater FRE from areas rich in carbonaceous formations such as limestone, although presumably the particular structure, depth, and layout of a deposition may also affect the extent of the carbonate exchange with the groundwater.

As a result of underwater photosynthesis, plants and algae become enriched with "old" carbon (with low ^{14}C content relative to the atmosphere), which then passes up the food chain to aquatic fauna (shellfish, fish, and mammals), and further to terrestrial animals that consume aquatic foods (including humans). The ^{14}C age of such samples thus appears older. It must be considered that the FRE value within the reservoir may vary depending on the type and age of the aquatic animal analyzed, subject to its specific habitat and diet (e.g. Fernandes et al. 2013). For example, because of the greater carbon exchange between the atmosphere and water, littoral fish and shellfish may be susceptible to FRE to a lesser extent than deep-water animals. There are also data on shifts of the FRE values over time as a result of changes in the hydrological structure, including geothermal conditions (e.g. Ascough et al. 2010) and depth of the reservoir (Geyh et al. 1998), or climatic conditions, leading to the melting of glaciers and/or permafrost and thus releasing a large amount of "old" carbon into the

Figure 1 Data on FROs for modern organisms and other material in the Eurasian Steppe from present and previous research. Numbers in parentheses correspond to those in Table 1. 1. Kharga Lake; 2. Kyzylkoi River; 3. Shat River (Sartyksu); 4. Nura River; 5. Syr-Darya River; 6. Karasuk Bay; 7. Yenisei River; 8. Edarma River; 9. Chuya River; 10. Katun River; 11. Lena River (Schulting et al., 2015); 12. Deed-Khulsun Lake (van der Plicht et al. 2016); 13. Volga River (van der Plicht et al. 2016), note that exact location is not available; 14. Tsimlyansk city (van der Plicht et al. 2016); 15. Serteya II site (Kulkova et al., 2015); 16. Podkumok River (Higham et al. 2010); 17. Caspian Sea, various locations (Olsson 1980; Arslanov and Tertychnaya 1983; Kuzmin et al. 2007); 18. Aral Sea, various locations (Kuzmin et al. 2007).

local reservoir (see discussion in Schulting et al. 2015; also Hågvar and Ohlson 2013; Hågvar et al. 2016).

Being highly variable in space and time, modern FREs may reach much larger ^{14}C offsets than observed in most marine surface water, often reaching thousands of ^{14}C years (Geyh et al. 1998; Hall and Henderson 2001; Ascough et al. 2010).

MATERIALS AND METHODS

To explore the extent of modern freshwater reservoir effects in the Eurasian Steppe, 12 samples of modern fish were analyzed for ^{14}C age and carbon (δ^{13}C) and nitrogen (δ^{15}N) stable isotopes (eight of which have been published in Svyatko 2016 and Svyatko et al. in press). Samples were sourced in various freshwater rivers and lakes of Siberia and Kazakhstan near archaeological sites in a region from 45.5°N, 61.4°E to 52.9°N, 111.9°E (Figure 1, Table 1). The geology of the region is very complex so that it is not possible to specify the source of old carbon for each water body. Fish species utilized included pike (*Esox lucius*), perch (*Perca sp.*), roach (*Rutilus rutilus*), burbot (*Lota lota*), crucian carp (*Carassius sp.*), and grayling (*Thymallus thymallus*).

The following water bodies have been analyzed (with sample-collection locations in parentheses):

- Kharga Lake (Eravninskiy Reg., Buryatia, Russia; 52°52′5′′N, 111°50′47′′E) is one of the largest lakes of the Eravno-Harginskaya Lake System (the total area of the latter is ca. 380 km^2). The system is fed by rainwater, snowmelt, and groundwater;

Table 1 Results of ^{14}C dating and stable isotope analysis of modern samples (with known collection year) from the Eurasian Steppe.

Nr	Lab ID	Material	Locality	Collection year	% coll.	$F^{14}C$	$F^{14}C$ atm mean	FRO (yr)	$\delta^{13}C$	$\delta^{15}N$	$C:N_{at}$
1	UBA-28386[1, a]	*Carassius sp.*	Kharga Lake, Eravninskiy Reg., Buryatia, Russia (52°52′5″N, 111°50′47″E)	2013	7.4	1.0372 ± 0.0036	1.0391 ± 0.002[b]	15 ± 35	−18.2	7.2	3.4
	UBA-28386-2[a]				10.7	1.0435 ± 0.0032	1.0391 ± 0.002[b]	−34 ± 28	−18.5	7.2	3.5
2	UBA-31310	*Perca sp.*	Kyzylkoi River, near Korzhar site, Kazakhstan (49°19′06.04″N, 73°30′55.62″E)	2014	8.6	1.0394 ± 0.0079	1.0391 ± 0.002[b]	2 ± 63	−28.9	11.9	3.9
3	UBA-27482[1]	*Rutilus rutilus*	Shat River (Sartyksu), Kazakhstan (50°38′20.51″N, 74°14′59.55″E)	2014	20.4	0.956 ± 0.0053	1.0391 ± 0.002[b]	670 ± 47	−30.6	6.8	3.6
4	UBA-27485[1]	*Carassius sp.*	Nura River (near Tegiszhol cemetery and Temirkash settlement), Kazakhstan (50°05′45.66″N, 72°45′53.14″E)	2014	16.1	0.9872 ± 0.0054	1.0391 ± 0.002[b]	412 ± 47	−26.6	15.6	3.9
5	UBA-29371[1]	n/a	Syr-Darya River (near Juvara site), Kazakhstan (45°30′58.0″N, 61°28′04.4″E)	2013?	15.8	1.0318 ± 0.0032	1.0391 ± 0.002[b]	57 ± 29	−12.9	10.0	3.2
6	UBA-29395[1]	*Esox lucius;* adult	Karasuk Bay, Minusinsk Basin, Russia (54°40′31.4″N, 90°49′47.0″E)	2007	13.7	0.9614 ± 0.0032	1.0564 ± 0.002	757 ± 31	−23.9	12.3	3.2
	UBA-29396[1]	*Esox lucius;* small		2007	9.1	0.976 ± 0.0033	1.0564 ± 0.002	636 ± 31	−26.1	12.6	3.5
	UBA-29397[1]	*Cyprinus carpio carpio*		2007	19.9	1.0041 ± 0.0032	1.0564 ± 0.002	408 ± 30	−24.2	8.9	3.1
7	UBA-29398[1]	*Cyprinus carpio*	Yenisei River (near Tepsei Mtn.), Minusinsk Basin, Russia (53.946669 N, 91.620614 E)	2007	13.1	1.0349 ± 0.0033	1.0564 ± 0.002	165 ± 30	−25.3	9.3	3.2
8	UBA-29646	*Lota lota*	Edarma River, Russia (53°56′48.0″N, 91°37′14.2″E)	2014?	7.5	0.9734 ± 0.0033	1.0391 ± 0.002[b]	525 ± 31	−19.5	14.0	3.0
9	UBA-31085	*Thymallus thymallus*	Chuya River, Kurai Basin, Russia (50°12′47.3″N, 87°53′22.7″E)	2015, Sept.	18.8	0.9064 ± 0.0038	1.0391 ± 0.002[b]	1097 ± 40	−27.7	7.9	3.8
10	UBA-31086	*Thymallus thymallus*	Katun River, Uimon Basin, Russia (50°11′35.0″N, 85°57′06.8″E)	2015, Sept.	13	0.967 ± 0.004	1.0391 ± 0.002[b]	578 ± 36	−24.0	5.8	3.8
11	OxA-V-2585-23[2]	*Esox lucius*	Lena River (near Ust–Kut), Irkutsk Obl, Russia (56°45′12.2″N, 105°39′12.0″E)	2002	n/a	0.84294 ± 0.0027	1.0786 ± 0.002	1981 ± 30[c]	−24.6	10.8	n/a
12	IGAN-3614[3,4]	*Cyprinus carpio*	Deed–Khulsun Lake, Caspian lowlands, Russia (46°17′50.6″N, 45°10′12.2″E)	2006	n/a	0.88295 ± 0.00547[c]	1.0612 ± 0.002	1477 ± 52[c]	n/a	n/a	n/a
	IGAN-3232[3,4]	*Cyprinus carpio*		2006	n/a	0.93266 ± 0.00578[c]	1.0612 ± 0.002	1037 ± 52[c]	−15.2	10.0	n/a
	IGAN-3708[4]	*Sediment*		2008	n/a	0.97784 ± 0.00848[c]	1.0521 ± 0.002	588 ± 71[c]	n/a	n/a	n/a
13	GrA-38097[4]	*Perca sp.*	Volga River, Russia	1915	n/a	0.86070 ± 0.00320 (1205 ± 30 BP)	104 ± 7 BP (from IntCal13)	1101 ± 31	n/a	n/a	n/a
14	GrA-45039[4]	Algae	Tsimlyansk city, Sukhaya Balka, Russia	2009	—	0.94199 ± 0.00351[c]	1.0494 ± 0.002	868 ± 34[c]	−20.5	n/a	—

15	SPb-14023[5]	*Squalius cephalus*	Serteya II site, Dvina–Lovat basin, Russia (55°40′58.0″N, 31°29′22.3″E)	n/a	n/a	0.953 ± 0.006	n/a	585 ± 69[d]	n/a	n/a	n/a
	SPb-1399[5]	*Nuphar lutea* root		n/a	—	0.988 ± 0.006	n/a	295 ± 68[d]	−25.5	—	—
	SPb-1400[5]	*Nuphar lutea* stem		n/a	—	99.9 ± 0.60	n/a	206 ± 67[d]	−25.9	—	—
	SPb-1401[5]	*Nuphar lutea* leaf		n/a	—	103.9 ± 0.60	n/a	−109 ± 66[d]	−25.3	—	—
16	OxA-14882[6]	Aquatic plant matter	Podkumok River, near Tereze town, Russia	2005, June	—	0.946 ± 0.003	1.0618 ± 0.002	928 ± 30	−19.8	8.4	—
	OxA-14861[6]	*Salmo trutta* (flesh)		2005, June	n/a	0.975 ± 0.003	1.0618 ± 0.002	685 ± 29	−26.7	11.7	n/a
	OxA-X-2139-18[6]	Water HCO_3		2005, June	—	0.660 ± 0.003	1.0618 ± 0.002	3819 ± 39	−19.7	—	—
17	U-4113[7]	*Phoca caspica*	Caspian Sea, various locations	1899	n/a	455 ± 50 BP	72 ± 7 BP	383 ± 50	−15.4	n/a	n/a
	n/a[8]	Mollusk shell		n/a	—	n/a	n/a	384 ± 59[d]	—	—	—
	AA-59489[9]	*Didacna crassa*		1953	—	410 ± 40 BP	0 ± 8 BP[d]	410 ± 41[d]	—	—	—
	AA-59487[9]	*Didacna ex gr. trigonoides*		ca. 1900	—	455 ± 30 BP	70 ± 7 BP[d]	385 ± 31[d]	—	—	—
	AA-59491[9]	*Didacna trigonoides*		ca. 1920	—	570 ± 30 BP	126 ± 8 BP[d]	444 ± 31[d]	—	—	—
	AA-59486[9]	*Didacna trigonoides*		ca. 1900	—	465 ± 35 BP	70 ± 7 BP[d]	395 ± 36[d]	—	—	—
18	AA-65490[9]	*Cerastoderma sp.*	Muynoq town, Aral Sea	1937	—	271 ± 49 BP	159 ± 6 BP[d]	112 ± 4[d]	—	—	—
	AA-65491[9]	*Cerastoderma sp.*	Kuzhetpes Island, Aral Sea	1936	—	433 ± 48 BP	159 ± 6 BP[d]	274 ± 4[d]	—	—	—
	AA-65492[9]	*Cerastoderma sp.*	Unknown location, Aral Sea	1944	—	276 ± 48 BP	159 ± 6 BP[d]	117 ± 48[d]	—	—	—

[1]Svyatko (2016); [2]Schulting et al. (2015); [3]Shishlina (2010); [4]van der Plicht et al. (2016); [5]Kulkova et al. (2015); [6]Higham et al. (2010); [7]Olsson (1980); [8]Arslanov and Tertychnaya (1983; as cited in Karpytchev [1993]); [9]Kuzmin et al. (2007).

[a]Note two aliquots of sample UBA-28386 were analyzed once with the lipid removal process (UBA-28386-2) and once without.

[b]$F^{14}C$ atm for the year 2012 was used (Levin et al. 2013).

[c]$F^{14}C$ and FRO were calculated from published BP ages using CALIBomb calibration program (http://calib.org/CALIBomb/).

[d]Values are taken from the source.

- Nura River (the sample was taken near Tegiszhol cemetery and Temirkash settlement, Kazakhstan; 50°05′45.66′′N, 72°45′53.14′′E) is the largest river of the Nura-Sarysuk basin at 978 km in length. It rises in the Kyzyktas Mountains and flows within the Kazakh Uplands into Tengiz Lake; the total area of the river basin is 58,100 km². Over 90% of the annual runoff of the river happens during the spring floods, while in the summer the upper streams of the river dry out;
- Kyzylkoi River (near Korzhar site, Kazakhstan; 49°19′06.04′′N, 73°30′55.62′′E) is a ca. 40-km-long tributary of the Sherubai-Nura River (tributary of the Nura River), fed mostly by snowmelt and groundwater;
- Shat River (also known as Sartyksu, Kazakhstan; 50°38′20.51′′N, 74°14′59.55′′E) is a tributary of the Bala-Shiderty River (flowing into Shiderty River); the total length of Shat and Bala-Shiderty is ca. 80 km. The river is fed mostly by snowmelt and groundwater;
- Syr-Darya River (near Juvara site, Kazakhstan; 45°30′58.0′′N, 61°28′04.4′′E) is 2212 km long, originating in the Tian Shan Mountains in Kyrgyzstan and eastern Uzbekistan and flows west and northwest through Uzbekistan and southern Kazakhstan to the remains of the Aral Sea;
- Yenisei River (near Tepsei Mountain, Minusinsk Basin, Russia; 53°56′48.0′′N, 91°37′14.2′′E), at 748 km long, this is the largest river system flowing to the Arctic Ocean; it originates in Mongolia and flows north to the Yenisei Gulf in the Kara Sea through a large part of central Siberia. The average depth is 14 m and the maximum depth is 24 m;
- Karasuk Bay (Minusinsk Basin, Russia; 54°40′31.4′′N, 90°49′47.0′′E) is 0.5 km wide and 8 km long; it was formed in the lower stream of the Kharasuk River, a tributary of the Yenisei River, in the late 1960s as a result of the construction of the Krasnoyarsk hydroelectric station;
- Edarma River (Russia; 58°45′46.6′′N, 102°34′47.9′′E) is a 153-km-long tributary of the Angara River;
- Katun River (Uimon Basin, Russia; 50°11′35.0′′N, 85°57′06.8′′E) is 688 km long and originates in the Katun glaciers on the southern slope of Belukha Mountain; its drainage basin covers 60,900 km^2; and
- Chuya River (Kurai Basin, Russia; 50°12′47.3′′N, 87°53′22.7′′E) is a 320-km-long tributary of the Katun River; its drainage basin covers 11,200 km^2. Left tributaries of the Chuya River originate from the glaciers of the North-Chuya Mountain Ridge.

Sample pretreatment and analysis were performed in the ^{14}CHRONO Centre for Climate, the Environment and Chronology (Queen's University, Belfast). Collagen extraction was based on the ultrafiltration method (Brown et al. 1988; Bronk Ramsey et al. 2004), which included the following steps:

1. Bone demineralization in 2% HCl, followed by MilliQ® ultrapure water wash;
2. Gelatinization in pH = 2 HCl at 58°C for 16 hr;
3. Filtration, using ceramic filter holders, glass filter flasks, and 1.2-μm glass microfiber filters;
4. Ultrafiltration using Vivaspin® 15S ultrafilters with MWCO 30 kDa; 3000–3500 rpm for 30 min; and
5. Freeze-drying; the dried collagen was stored in a desiccator.

Bone collagen stable carbon and nitrogen isotopes were measured in duplicate on a Thermo Delta V isotope ratio mass spectrometer (IRMS) coupled to a Thermo Flash 1112 elemental analyzer (EA) peripheral. The measurement uncertainty (1σ) of $\delta^{13}C$ and $\delta^{15}N$ based on 6–10 replicates of 7 archaeological bone collagen samples was 0.22‰ and 0.15‰, respectively. The reference standards used were IA-R041 L-alanine, IAEA-N-2 ammonium sulphate, IA-R001 wheat flour, IAEA-CH-6 sucrose, and nicotinamide. Results are reported using the delta convention relative to international standards: VPDB for $\delta^{13}C$ and AIR for $\delta^{15}N$ (Hoefs 2009). No lipid removal was applied to the majority of fish samples as the primary aim of their analysis was to assess the freshwater reservoir effect in the area and the retention of lipids in the sample does not affect its ^{14}C age or $\delta^{15}N$ ratios (e.g. Guiry et al. 2016). Only one sample, UBA-28386-2, was subjected to the lipid removal process following Bligh and Dyer (1959) as well as being analyzed without lipid removal.

For the accelerator mass spectrometry (AMS) ^{14}C measurements, prepared bone collagen samples were sealed under vacuum in quartz tubes with an excess of CuO and combusted at 850°C. The CO_2 was converted to graphite on an iron catalyst using a zinc reduction method (Slota et al. 1987). The pressed graphite "target" was then measured on a 0.5 MV National Electrostatics Compact AMS. The sample $^{14}C/^{12}C$ ratio was background corrected and normalized to the HOXII standard (SRM 4990C; National Institute of Standards and Technology). The $^{14}C/^{12}C$ ratio corrected for isotopic fractionation using the AMS-measured $\delta^{13}C$, is equivalent to fraction modern ($F^{14}C$; Reimer et al. 2004). The ^{14}C age and 1σ were calculated from $F^{14}C$ using the Libby half-life (5568 yr) following the conventions of Stuiver and Polach (1977). The ^{14}C ages were calibrated using the Calib 7.0 program (Stuiver et al. 2013) and the IntCal13 calibration curve (Reimer et al. 2013) where appropriate.

Freshwater reservoir offsets (FRO) were calculated as a difference in the ^{14}C ages between the fish and atmosphere. Atmospheric ^{14}C age and ^{14}C age for the modern fish samples (conventionally given as > modern) were calculated using the following equation: $^{14}C\ \text{age} = -8033 \times \ln(F^{14}C)$. $F^{14}C_{atm}$ for various years were taken as an average of the monthly $^{14}C_{atm}$ measurements from Levin et al. (2013). For samples collected in 2012 and later, average $F^{14}C_{atm}$ for the year 2012 was used. ^{14}C age uncertainties were calculated using the following formula: $\sigma^{14}C = -8033 \times \ln(F^{14}C + \sigma F^{14}C - (-8033 \times \ln(F^{14}C))$ for each sample. FRO uncertainty was calculated using $\sigma FRO = \sqrt{\sigma a^2 + \sigma b^2}$, where σa and σb are ^{14}C age uncertainties for fish samples and atmosphere. For the pre-1950 sample the atmospheric value was taken from IntCal13 (Reimer et al. 2013).

RESULTS AND DISCUSSION

The results, along with those from previous research, are presented in Table 1 and Figures 1 and 2 and are available online at http://chrono.qub.ac.uk/FRE/. In most samples analyzed, the collagen content varied between 7.4 and 20.4%, and the atomic C:N ($C{:}N_{at.}$) ratio varied between 3.0 and 3.9 (although we cannot exclude the potential admixture of lipids in the collagen samples).

The resulting data, combined with the existing published results, allow us to make a number of observations:

Freshwater Reservoir Effects

1. FREs are present in the majority of the areas analyzed, apart from Kharga Lake (FRO = 15 ± 35 ^{14}C yr) and Kyzylkoi River (FRO = 2 ± 63 ^{14}C yr)—these two reservoirs need further analysis of modern aquatic fauna to confirm the absence of any offset.

The apparent lack of the FRO in these waterbodies is possibly due to specifics of local geology (such as the absence of old ^{14}C-free bedrock exposures). For other reservoirs, the particular sources of old carbon are not clear but might include meltwater from permafrost such as in the case of the Katun and Chuya Rivers.

2. The FROs detected in present study are highly variable, ranging between 15 ± 35 and 2 ± 63 ^{14}C yr (i.e., no offset) to 1097 ± 40 ^{14}C yr in various locations. Even higher offsets have been observed in previous research for the Caspian lowlands (1477 ± 52 and 1037 ± 52 ^{14}C yr; Shishlina 2010; van der Plicht et al. 2016), Upper Lena River basin (Lake Baikal region, 1981 ± 30 ^{14}C yr; Schulting et al. 2015), and Podkumok River (Karachay-Cherkess Republic, Russia, 3819 ± 39 ^{14}C yr; Higham et al. 2010).
3. The FROs also vary within single reservoirs between different species and different sizes/ages of fish. As a particular example, in the Minusinsk basin of southern Siberia, FRO values vary both between different reservoirs (with the tributary Karasuk having a higher offset compared to that of the main Yenisei River) and between species and fish of different age within the reservoir depending on their diet (the two pikes from the Karasuk Bay having the highest offsets, and the larger pike being ca. 120 ^{14}C yr "older" than the smaller one).

Many of the analyzed Eurasian Steppe reservoirs clearly contain depleted carbon; however, its particular sources remain unclear and are beyond the scope of the current study.

Stable Isotope Results

As no lipid removal procedure was applied to the majority of samples, their δ^{13}C values potentially reflect combined collagen and lipid carbon isotopic signal. Lipids are known to be significantly lower in δ^{13}C—recent research has demonstrated up to 5‰ increase in δ^{13}C of fish-bone samples subjected to lipid removal (Guiry et al. 2016). It must also be acknowledged that for modern aquatic samples, the isotopic values can be affected by present day agricultural and industrial activities in the area.

1. A variety of both δ^{13}C and δ^{15}N values can be observed in modern freshwater fish from the Eurasian Steppe. The majority of samples (with the exception of four samples from Kharga Lake and Edarma River in western Siberia, Deed-Khulsun Lake in Caspian lowlands and Syr-Darya River in Kazakhstan) apparently reflect C_3 ecology of local reservoirs (though again, it should be noted here that the potential presence of lipids may have biased the resulting δ^{13}C towards lower values). The higher δ^{13}C values of the four samples above could reflect modern agricultural and industrial practices in the areas, and, as only single samples have been analyzed for these reservoirs, further isotopic measurements of additional specimens is needed to verify this. However, notably, a number of modern fish from the western Siberia regions, including Lake Baikal, have also previously demonstrated elevated δ^{13}C levels (e.g. Katzenberg and Weber 1999; Weber et al. 2011 and others). Nitrogen isotope ratios of the fish appear related primarily to the diet of different species, with predatory species such as pike, perch, salmon, and burbot (Figure 2, filled symbols) demonstrating the highest δ^{15}N. Surprisingly, the highest δ^{15}N can be seen in a crucian carp from the Nura River (Kazakhstan). Whether this reflects the diet of the fish, the river's natural state, or is the effect of modern agricultural or industrial activities, cannot be said.
2. No general relationship has been observed between FROs and either δ^{13}C or δ^{15}N values of the samples (in both cases $p > 0.05$). To explore any possible local trends in this regard, FROs in individual regions would need to be explored in greater detail involving larger number of modern aquatic samples.

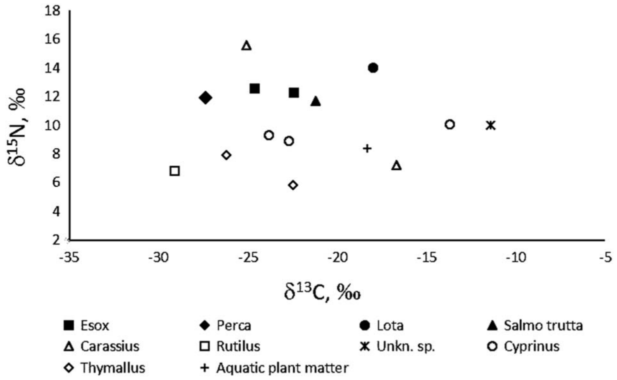

Figure 2 Stable isotope results of modern freshwater fish (n = 15, including three samples from previous research) and plants (n = 1) analyzed. Data on fish and plant matter from previous research were taken from Higham et al. (2010), Schulting et al. (2015), and Shishlina (2010). As the *Salmo trutta* sample was taken from flesh, 4‰ was added to its δ^{13}C value to make it more comparable to bone collagen measurements.

SUMMARY AND CONCLUSION

This research represents the first broad-scale study of FRE across the Eurasian Steppe region, specifically focused on modern samples. The aim of this work was not only to demonstrate the widespread variability of FREs in the territory of Russia and Kazakhstan, but also to draw the attention of specialists working in the area to the necessity of taking into account this important factor in ^{14}C dating human and some faunal remains from archaeological sites.

In calculating the average FRO for particular regions or archaeological cultures, several factors need to be considered. Firstly, as FROs are extremely diverse geographically, an average offset should be calculated for a specific area (or even a particular reservoir), rather than for entire cultures/populations as the distribution of a population can span multiple regions with various geological characteristics. Secondly, for terrestrial animals (including humans), consuming aquatic foods, the degree of the offset for each individual will directly depend on the proportion of aquatic food in the diet, and further stable isotope analysis is essential to assess the latter (e.g. see Schulting et al. 2014). For example, the difference between the paired human-herbivore bone samples from the Mesolithic to the Early Bronze Age in the upper Lena River basin ranges from 255 to 1010 ^{14}C yr (Schulting et al. 2015). For the aquatic samples themselves (fish and shellfish), the extent of the FRO, as mentioned above, is directly linked to the diet and habitat of the animal and can vary significantly between individuals. Thus, at the moment there is a need for further research to demonstrate the appropriateness of the average FRO correction for different populations.

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