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#### Contents lists available at ScienceDirect

# Data in Brief





#### Data Article

# Carbon, nitrogen and sulphur isotope data of archaeological fish and mammal bone collagen from Lithuania



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Stable isotope analysis
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Palaeodiet
Palaeoeconomy
Mesolithic
Subneolithic
Neolithic
Bronze age

#### ABSTRACT

Until relatively recently, stable sulphur isotope analysis of bone collagen was seldom undertaken in bioarchaeological research. With increasing frequency, its application has proven useful in reconstructing palaeodiets and palaeoecologies, as well as identifying potential migration and mobility patterns. Here, sulphur ( $\delta^{34}$ S) isotope analysis, together with carbon ( $\delta^{13}$ C) and nitrogen ( $\delta^{15}$ N), was performed on six fish and 34 mammal bone collagen samples from 14 prehistoric sites in Lithuania dating from the Late Mesolithic (ca. 7000–5000 cal BC) to the Late Bronze Age (ca. 1100–500 cal BC). We present the first  $\delta^{34}$ S data from Lithuania, including coupled  $\delta^{13}$ C and  $\delta^{15}$ N data, offering a crucial dataset for future research to explore spatial and temporal variability in the region and beyond.

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## Specifications Table

Subject	Social Sciences - Archaeology
Specific subject area	Archaeology
	Bioarchaeology
	Stable isotope analysis
	Bone collagen
	Carbon
	Nitrogen
	Sulphur
	Palaeodiet
	Palaeoeconomy
Type of data	Table
	Figure
How data were	Carbon, nitrogen and sulphur stable isotope measurements were obtained by
acquired	Isotope Ratio Mass Spectrometry (IRMS). A Costech Elemental Analyser (ECS
	4010) connected to a Thermo Scientific Delta V Advantage IRMS system was
	used to generate the $\delta^{13} C$ and $\delta^{15} N$ data. However, $\delta^{34} S$ data was generated
	using an IsoLink connected to a Thermo Scientific Delta V Plus IRMS.
Data format	Raw
Description of data collection	The bone samples were demineralised, and the collagen was extracted
	following a standard modified Longin method [1,2,3,4].
Data source location	Institution: Durham University
	City: Durham
	Country: United Kingdom
	Latitude and longitude for collected samples/data: Daktariškė 5
	(55°47′54.82′'N, 22°23′15.68′'E), Donkalnis (55°48′26.95"N, 22°25′19.77"E),
	Kretuonas 1B (55°15′38.92"N, 26°5′58.18"E), Plinkaigalis (55°24′38.27"N,
	23°38′48.67″E), Spiginas (55°46′3.93″N, 22°25′2.24″E), Šventoji 1 (56°1′1.92′'N,
	21°5′21.85′E), Šventoji 2 (56°0′53.75′N, 21°5′10.22′E), Šventoji 3
	(56°0′57.18′'N, 21°5′13.20′'E), Šventoji 4 (56°0′53.23′'N, 21°5′5.87′'E), Šventoji
	23 (56°0′1.68"N, 21°5′23.53"E), Šventoji 26 (56°0′8.08′'N, 21°5′27.29′'E),
	Šventoji 43 (55°58′50.43"N, 21°5′20.5"E), Turlojiškė (54°21′45.78"N,
	23°18′3.47″E) and Žemaitiškė 2 (55°15′36.81′′N, 26°6′29.66′′E)
Data accessibility	This dataset is deposited in IsoArcH [5] (www.isoarch.eu) with the following
	Digital Object Identifier (DOI): https://doi.isoarch.eu/doi/2022.003
	Data identification number: 10.48530/isoarch.2022.003
	Data is available under the Creative Commons BY-NC-SA 4.0 license.

#### Value of the Data

- These data represent the first  $\delta^{34}$ S values derived from prehistoric material from Lithuania, and can be utilised for future research to examine temporal and spatial variability in the region.
- These isotopic data will be useful to archaeologists, especially bioarchaeologists, examining palaeodiets and palaeoecologies dating to the Late Mesolithic, Subneolithic, Neolithic and Bronze Age throughout Europe.
- These data supplement the limited number of  $\delta^{13}C$  and  $\delta^{15}N$  data obtained previously from prehistoric fish bone collagen [see 6].
- These data can be compared with other data from prehistoric sites in Lithuania and beyond.

## 1. Objective

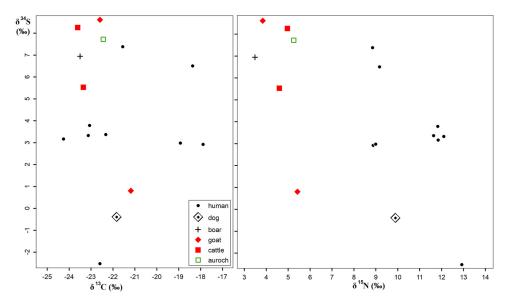
For more than 30 years, stable isotope analysis has been successfully employed in palaeodietary reconstructions. Its application to identify palaeomobility has, however, only recently gathered momentum. To determine the origin(s) of potential migrants and evaluate the extent of mobility amongst prehistoric communities in Lithuania, associated with the Comb Ware and Corded Ware cultures, the IZOMOB project (2020-2022) was launched. Sulphur ( $\delta^{34}$ S), strontium ( $^{87}$ Sr)<sup>86</sup>Sr) and oxygen ( $\delta^{18}$ O) isotope analyses was undertaken on modern soil and water samples as well as a range of modern and prehistoric faunal and floral remains. This dataset is one output of that project.

#### 2. Data Description

These data include carbon ( $\delta^{13}$ C), nitrogen ( $\delta^{15}$ N) and sulphur ( $\delta^{34}$ S) stable isotope values extracted from fish and mammal bone samples. A number of taxa were sampled, including aurochs (*Bos primigenius*), domestic cattle (*Bos taurus*), common bream (*Abramis brama*), domestic dog (*Canis familiaris*), Eurasian beaver (*Castor fiber*), goat (*Capra* sp.), harp seal (*Pagophilus groenlandicus*), human (*Homo sapiens*), northern pike (*Esox lucius*) and wild boar (*Sus scrofa*), representing a range of behavioural life histories and habitat uses. The specimens date to the Late Mesolithic (*ca.* 7000–5000 cal BC), Subneolithic (*ca.* 5000–2900 cal BC), Neolithic (*ca.* 2900–1800 cal BC), Early Bronze Age (*ca.* 1800–1100 cal BC) and Late Bronze Age (*ca.* 1100–500 cal BC). The samples were derived from 14 prehistoric sites (i.e., Daktariškė 5, Donkalnis, Kretuonas 1B, Plinkaigalis, Spiginas, Šventoji 1, Šventoji 2, Šventoji 3, Šventoji 4, Šventoji 23, Šventoji 26, Šventoji 43, Turlojiškė and Žemaitiškė 2) in Lithuania (Fig. 1). The localities are represented by burial grounds, fishing stations, ritual depositions and settlement sites which have differing research histories. Excavations took place during the 20<sup>th</sup> and 21<sup>st</sup> centuries [see 6–11]. Samples were collected by one of us (GyP) and then submitted for isotopic analysis to the Stable Isotope



Fig. 1. Locations of the 14 prehistoric sites in Lithuania from where the samples were obtained.



**Fig. 2.** Carbon  $(\delta^{13}C)$ , nitrogen  $(\delta^{15}N)$  and sulphur  $(\delta^{34}S)$  stable isotope data obtained on fish and mammal bone collagen.

Biogeochemistry Laboratory (SIBL), managed and operated by another one of us (DRG) in the Department of Earth Sciences, Durham University.

In total, 22 animal and 18 human bone samples were prepared for analysis. Of these, 27 produced sufficient quantities of well-preserved collagen for carbon and nitrogen stable isotope analysis, yielding atomic C:N ratios within the acceptable range of between 2.9 and 3.6 according to DeNiro [4]. A total of 16 samples yielded sufficient quantities of well-preserved collagen for sulphur stable isotope analysis, producing atomic C:S and N:S ratios within the acceptable ranges of between 600  $\pm$  300 and 200  $\pm$  100, respectively, according to Nehlich and Richards [12]. Four samples, however, yielded only enough collagen for a single sulphur measurement, consequently carbon and nitrogen stable isotope data from a previous study was utilised [8]. These data also yielded atomic C:N ratios which were within the acceptable range, indicating a low likelihood of diagenesis [4]. Stable isotope measurements were performed in the Stable Isotope Biogeochemistry Laboratory (SIBL) at Durham University using a Thermo IsoLink coupled to a Thermo Scientific Delta V Plus Advantage isotope ratio mass spectrometer. Sulphur isotope ratios are reported in standard delta ( $\delta$ ) notation in per mil ( $\infty$ ) relative to the VCDT scale. Correction of  $\delta^{34}$ S was performed using four international standards (IAEA-S-1, IAEA-S-2, IAEA-S-3, NBS 127): this provided a linear range in  $\delta^{34}$ S between -32.5% and +22.6%. Analytical uncertainty of  $\delta^{34}$ S is typically  $\pm 0.2\%$  for replicate analyses of the international standards. Total sulphur is determined as part of the isotopic analysis (i.e., total peak area) using an internal standard, sulphanilamide (18.6196% sulphur). Further details on analytical procedures, standards and errors for carbon and nitrogen are reported in Walser et al. [13].

We obtained a grand total of 27  $\delta^{13}$ C and  $\delta^{15}$ N values. In addition, 20  $\delta^{34}$ S values were obtained, making it the largest dataset of  $\delta^{34}$ S values from prehistoric sites in Lithuania. The  $\delta^{13}$ C and  $\delta^{15}$ N values are plotted against the  $\delta^{34}$ S values in Fig. 2. In Fig. 3 the dataset is compared with previously published data from Lithuania. Table 1 presents the data per sample, including sample number, site, grave number or individual, period, species or taxon, skeletal element, ontogentic age, context, excavation campaign and/or find number, %N,  $\delta^{15}$ N, %C,  $\delta^{13}$ C, %S,  $\delta^{34}$ S, C:N, C:S and N:S. Summary statistics for the various groups and species or taxon are shown in Table 2. This dataset is deposited in IsoArcH [5] (www.isoarch.eu) with the following

Table 1
Fish and mammal bone samples and stable isotope data. Note that the data has been sorted alphabetically according to the site. Although the C:S ratio for one sample (HUM-08), a human humerus from Kretuonas 1B (Grave No. 3), was unacceptable according to the criteria defined by Nehlich and Richards [12], both the C:N and N:S ratios were within the acceptable ranges defined by DeNiro [4] and Nehlich and Richards [12]. As such, it was included here. Blank, no collagen preserved or no data. \*, mean of stable isotope data from NAU and UOY [see 8]. \*\*, stable isotope data from NAU only [see 8].

Sample no.	no. Site, grave Period Species or taxon Skeletal element, ontogenetic %N no./individual age, context, excavation campaign, find no.		%N	δ <sup>15</sup> N (‰)	%C	δ <sup>13</sup> C (‰)	%S	δ <sup>34</sup> S (‰)	Atomic C:N	Atomic C:S	Atomic N:S		
AN-01	Daktariškė 5	Subneolithic	Esox lucius	Cleithrum, sin., 60-70 cm in length, 2016 year, No. 2557					0.3	-0.1			
AN-02	Daktariškė 5	Subneolithic- Early Bronze Age	Canis familiaris	Radius, sin.	14.29	9.89	40.08	-21.84	0.31	-0.39	3.27	350.64	107.22
AN-03	Daktariškė 5	Subneolithic- Early Bronze Age	Capra sp.	Mandible, sin., adult	13.60	3.84	38.29	-22.59	0.26	8.61	3.28	395.47	120.42
AN-04	Daktariškė 5	Subneolithic- Early Bronze Age	Bos taurus	Femur, sin.	14.67	4.61	41.69	-23.35	0.27	5.53	3.31	407.42	122.93
AN-05	Daktariškė 5	Subneolithic- Early Bronze Age	Bos primigenius	Metacarpus, sin., 2016 year, No. 1797	14.90	5.26	41.91	-22.44	0.25	7.72	3.28	443.83	135.29
AN-06	Daktariškė 5	Subneolithic	Sus scrofa	Mandible, 10 months-year, 2016 year, No. 569	10.58	3.49	32.32	-23.49	0.18	6.94	3.56	482.36	135.36
AN-07	Daktariškė 5	Subneolithic- Early Bronze Age	Castor fiber	Fibula, dex., young, 2016 year, No. 1172	13.79	3.69	38.31	-22.02			3.24		
HUM-04	Donkalnis, Grave No. 1	Subneolithic	Homo sapiens	Femur diaphysis, 20-25 year-old female	13.84	11.84	37.99	-24.26	0.20	3.17	3.20	502.10	156.81
HUM-05	Donkalnis, Grave No. 6	Subneolithic	Homo sapiens	Fibula diaphysis, 35-40 year-old female	15.48	11.63	42.13	-22.32	0.21	3.37	3.17	545.11	171.69
HUM-06	Donkalnis, Grave No. 7	Late Subneolithic	Homo sapiens	Fibula diaphysis, >45 year-old male	9.65	10.61	28.29	-21.74			3.42		
HUM-07	Donkalnis, 5 <sup>th</sup> individual	Late Mesolithic	Homo sapiens	J245D, ~7 year-old individual					0.21	4.18			
AN-08	Kretuonas 1B	Subneolithic- Neolithic	Esox lucius	Precaudal vertebra, 80-100 cm in length									
AN-09	Kretuonas 1B	Subneolithic- Neolithic	Bos primigenius	2nd phalanx									

Table 1 (continued)

Sample no.	Site, grave no./individual	Period	Species or taxon	Skeletal element, ontogenetic age, context, excavation campaign, find no.	%N	δ <sup>15</sup> N (‰)	%C	δ <sup>13</sup> C (‰)	%S	δ <sup>34</sup> S (‰)	Atomic C:N	Atomic C:S	Atomic N:S
AN-10	Kretuonas 1B	Subneolithic- Neolithic	Sus scrofa	Astragalus, sin.	8.48	4.84	25.10	-23.31			3.45		
AN-11	Kretuonas 1B	Subneolithic- Neolithic	Castor fiber	Tibia, dex., young	12.41	3.29	36.85	-22.72			3.46		
AN-12	Kretuonas 1B	Subneolithic- Neolithic	Cyprinidae, cf. Abramis brama	1 x precaudal vertebra; 3 x caudal vertebrae									
HUM-08*	Kretuonas 1B, Grave No. 3	Subneolithic	Homo sapiens	Humerus diaphysis, 50-55 year-old male	13.90	11.80	38.70	-22.80	0.11	5.97	3.20	945.43	291.18
HUM-17	Plinkaigalis, Grave No. 241	Neolithic	Homo sapiens	Femoral distal epiphysis, 50-55 year-old female	10.36	8.85	29.24	-21.55	0.22	7.39	3.29	360.77	109.64
HUM-18	Plinkaigalis, Grave No. 242	Neolithic	Homo sapiens	Upper dex. molar (M2), root, >40 year-old female									
HUM-01	Spiginas, Grave No. 1	Subneolithic	Homo sapiens	Femur diaphysis, 35-45 year-old male	10.21	11.83	29.52	-23.07	0.18	3.79	3.37	429.74	127.39
HUM-02	Spiginas, Grave No. 3	Late Mesolithic	Homo sapiens	Fibula diaphysis, indeterminate female	14.84	12.10	41.96	-23.13	0.24	3.33	3.30	469.20	142.24
HUM-03	Spiginas, Grave No. 4	Late Mesolithic	Homo sapiens	30-35 year-old female	15.02	12.92	41.78	-22.59	0.22	-2.53	3.24	509.59	157.15
AN-16	Šventoji 1	Subneolithic- Neolithic	Canis familiaris	Mandible, 1968 year	12.62	14.46	35.89	-16.38			3.32		
AN-18	Šventoji 2	Subneolithic- Neolithic	Sus scrofa	Femur, sin., adult	14.03	4.47	40.43	-21.47			3.36		
AN-19	Šventoji 2	Subneolithic- Neolithic	Castor fiber	Femur, sin., young	14.37	4.02	41.32	-22.56			3.35		
AN-20	Šventoji 3	Subneolithic- Neolithic	Pagophilus groenlandicus	Os temporale, sin., adult, 1972 year	14.46	9.51	40.80	-23.33			3.29		
AN-14	Šventoji 4	Subneolithic	Esox lucius	Dentary, sin., 90-100 cm in length, 2014 year, No. 1514 (1038)									
AN-15	Šventoji 4	Subneolithic	Abramis brama	Preopercular, subopercular, opercular, 2014 year, No's. 1218-1227	12.50	7.92	36.83	-23.94			3.44		
AN-17	Šventoji 4	Subneolithic- Neolithic	Bos primigenius	Maxilla, 2003 year	13.68	5.01	38.21	-22.66			3.26		

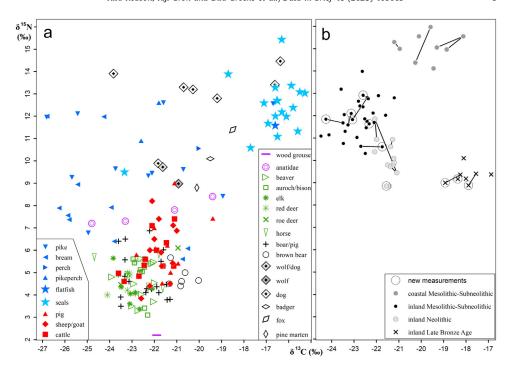
(continued on next page)

Table 1 (continued)

Sample no.	Site, grave no./individual	Period	Species or taxon	Skeletal element, ontogenetic age, context, excavation campaign, find no.	: %N	δ <sup>15</sup> N (‰)	%C	δ <sup>13</sup> C (‰)	%S	δ <sup>34</sup> S (‰)	Atomic C:N	Atomic C:S	Atomic N:S
HUM-09	Šventoji 23, single bone No. 1	Subneolithic	Homo sapiens	13-18 year-old individual									
HUM-10	Šventoji 23, single bone No. 2	Subneolithic	Homo sapiens	20-30 year-old individual									
HUM-12	Šventoji 26, single bone No. 1	Subneolithic	Homo sapiens	Femur diaphysis									
HUM-11	Šventoji 43, single bone No. 2	Subneolithic	Homo sapiens	Lower dex. molar									
AN-21	Turlojiškė	Late Bronze Age	Esox lucius	Dentary, dex., 70-80 cm in length, 1998 year	14.28	12.56	40.38	-16.70			3.30		
AN-22	Turlojiškė	Late Bronze Age	Capra sp.	Humerus, dex., >11-13 months, 1998 year, IV, I trench, C-11	13.39	5.42	36.31	-21.18	0.29	0.80	3.16	339.97	107.52
HUM-13	Turlojiškė, Grave No. 3	Late Bronze Age	Homo sapiens	Ulna diaphysis, 25-30 year-old male	14.08	9.17	39.03	-18.36	0.25	6.51	3.23	410.15	126.83
HUM-14**	Turlojiškė, Grave No. 4	Late Bronze Age	Homo sapiens	20-25 year-old male	15.40	9.40	43.40	-16.90	0.25	6.99	3.30	466.07	141.81
HUM-15	Turlojiškė, Grave No. 5	Late Bronze Age	Homo sapiens	"IIv, 6 perk.", 1999 year	14.09	8.88	40.00	-17.88	0.28	2.92	3.31	377.44	114.00
HUM-16	Turlojiškė, Grave No. 6	Late Bronze Age	Homo sapiens	"II v, 7 perk.", 1999 year	13.05	9.00	37.55	-18.92	0.28	2.98	3.36	356.75	106.30
AN-13	Žemaitiškė 2	Early Bronze Age	Bos taurus	M2	12.99	4.97	36.44	-23.60	0.29	8.26	3.27	340.39	104.03

**Table 2**Summary statistics for the various groups and species (or taxon). Note that the human data has been disaggregated according to site. Standard deviations have only been calculated for N > 2. Blank, no data.

Group	N	Mean δ <sup>13</sup> C (‰)	Max δ <sup>13</sup> C (‰)	Min δ <sup>13</sup> C (‰)	Range δ <sup>13</sup> C (‰)	N	Mean δ <sup>15</sup> N (‰)	Max δ <sup>15</sup> N (‰)	Min δ <sup>15</sup> N (‰)	Range δ <sup>15</sup> N (‰)	N	Mean δ <sup>34</sup> S (‰)	Max δ <sup>34</sup> S (‰)	Min δ <sup>34</sup> S (‰)	Range δ <sup>34</sup> S (‰)
Homo sapiens	12	-21.13	-16.90	-24.26	7.36	12	10.67	12.92	8.85	4.07	12	4.01	7.39	-2.53	9.92
Donkalnis	3	-22.77	-21.74	-24.26	2.52	3	11.36	11.84	10.61	1.23	3	3.57	4.18	3.17	1.01
Kretuonas 1B	1	-22.80				1	11.80				1	6.00			
Plinkaigalis	1	-21.55				1	8.85				1	7.39			
Spiginas	3	-22.93	-22.59	-23.07	0.48	3	12.28	12.92	11.83	1.09	3	1.53	3.79	-2.53	6.32
Turlojiškė	4	-18.02	-16.90	-18.92	2.02	4	9.11	9.40	8.88	0.52	4	4.85	6.99	2.92	4.07
Semi-aquatic herbivores	3	-22.07	-22.72	-22.02	-0.70	3	5.85	4.02	3.29	0.73					
Castor fiber	3	-22.22	-22.72	-22.02	-0.70	3	6.50	4.02	3.29	0.73					
Terrestrial herbivores	6	-21.98	-21.18	-23.60	2.42	6	6.09	5.42	3.84	1.58	5	6.31	8.61	0.80	7.81
Bos primigenius	2	-22.28	-22.44	-22.66	0.22	2	5.88	5.26	5.01	0.25	1	7.72			
Bos taurus	2	-22.99	-23.35	-23.60	0.25	2	4.31	4.97	4.61	0.37	2	7.11	8.26	5.53	2.73
Capra sp.	2	-21.98	-21.18	-22.59	1.41	2	6.09	5.42	3.84	1.58	2	6.31	8.61	0.80	7.81
Marine carnivores	1	-23.33				1	9.51								
Pagophilus groenlandicus	1	-23.33				1	9.51								
Freshwater fish	2	-21.00	-16.70	-23.94	7.24	2	8.28	12.56	7.92	4.64	1	-0.13			
Abramis brama	1	-23.94				1	7.92								
Esox lucius	1	-16.70				1	12.56				1	-0.13			
Omnivores	5	-22.29	-16.38	-23.49	7.11	5	5.83	14.46	3.49	10.97	2	5.68	6.94	-0.39	7.33
Canis familiaris	2	-22.74	-16.38	-21.84	5.46	2	5.42	14.46	9.89	4.57	1	-0.39			
Sus scrofa	3	-22.01	-21.47	-23.49	2.02	3	6.42	4.84	3.49	1.35	1	6.94			



**Fig. 3.** Comparison of the carbon ( $\delta^{13}$ C) and nitrogen ( $\delta^{15}$ N) stable isotope data for fauna (a) and humans (b) obtained with previously published data from Lithuania [6,8,9,16]. Connecting lines, measurements made on different skeletal elements of the same individual. This variation likely reflects differences in the bone turnover rates between skeletal elements rather than inter-instrument variation between institutions [see 17].

Digital Object Identifier (DOI): https://doi.isoarch.eu/doi/2022.003; data identification number: 10.48530/isoarch.2022.003; data is available under the Creative Commons BY-NC-SA 4.0 license.

The humans (n=12) had a broad range of  $\delta^{13}$ C (-24.3% to -16.9%),  $\delta^{15}$ N (+8.9% to +12.9%) and  $\delta^{34}$ S (-2.5% to +7.4%) values. There were two groups with one indicating the consumption of freshwater derived protein and the other demonstrating the consumption of C<sub>4</sub> foodstuffs, including millet [see 8], agreeing with their chronological affiliation (i.e., hunter-gatherer-fishers in the former and agriculturalists in the latter). Since all human  $\delta^{34}$ S values were below +10.0%, which is considered a minimum for the impacts of the sea spray affect [see 18], they indicate inland residency. This is perhaps not unexpected since the site closest to the coast yielding  $\delta^{34}$ S values was 86 km away (i.e., Donkalnis), while the impacts of the sea spray affect are known up to ca, 30 km inland [18].

The semi-aquatic herbivores (n=3), namely the Eurasian beaver, had a narrow range of  $\delta^{13}$ C (-22.7‰ to -22.0‰) and  $\delta^{15}$ N (+3.3‰ to +4.0‰) values, agreeing with their habitat use.

Similarly, the terrestrial herbivores (n=6), including aurochs, domestic cattle and goat, had a narrow range of  $\delta^{13}$ C (-23.6% to -21.2%) and  $\delta^{15}$ N (+3.8% to +5.4%) values, though variable  $\delta^{34}$ S (n=5; +0.8% to +8.6%) values. Despite this, taken together these data indicate that they were living in somewhat similar environments unaffected by the canopy effect [19] and/or a contribution of sea spray. Similarly, the site nearest the coast yielding  $\delta^{34}$ S values was some 84 km away (i.e. Daktariškė 5).

The one marine carnivore in the dataset, a harp seal from Šventoji 3, had a  $\delta^{13}$ C value of -23.3‰ and a  $\delta^{15}$ N value of +9.5‰. These data indicate residency in a  $^{13}$ C-enriched environment such as the Šventoji Palaeolake directly in front of the site or the nearby Baltic Sea [see 14,15].

Several common bream remains from Šventoji 4 were combined to ensure that enough collagen could be extracted for stable isotope analysis. The  $\delta^{13}$ C (-23.9‰) and  $\delta^{15}$ N (+7.9‰) values indicate residency in a similar environment to the harp seal from Šventoji 3. In contrast, a northern pike dentary from the wetland site of Turlojiškė had a  $\delta^{13}$ C value of -16.7‰ and a  $\delta^{15}$ N value of +12.6‰, which despite being notably higher than the common bream, indicated freshwater residency [see 20]. Only one fish bone, a northern pike cleithrum from Daktariškė 5, yielded sufficient quantities of collagen for a single sulphur measurement (-0.1‰), which likewise demonstrated residency in a freshwater environment.

While the two dog bones had divergent  $\delta^{13}$ C (-21.8‰ and -16.4‰) and  $\delta^{15}$ N values (+9.9‰ and +14.5‰), it is possible that both either consumed resources from a freshwater environment or perhaps from a fresh water in the case of the former, and an intermediary waterbody enriched in  $^{13}$ C, such as the Baltic Sea [14,15], in the latter. The one sulphur measurement (-0.4‰) obtained from a dog from Daktariškė 5 demonstrated inland residency.

The wild boars (n=3) had a narrow range of  $\delta^{13}C$  (-23.5% to -21.5%) and  $\delta^{15}N$  (+3.5% to +4.8%) values. Despite being derived from three different sites (i.e., Daktariškė 5, Kretuonas 1B and Šventoji 2), these data indicate little omnivory. The single sulphur measurement (+6.9%) demonstrated inland residency.

## 3. Experimental Design, Materials and Methods

A range of species (or taxa) from each assemblage were selected for analysis. This ensured that the same individual was not sampled more than once. With regards to the human bone samples, all individuals were derived from different burials or in the case of the loose human bones from Šventoji 23 were clearly separated by ontogenetic age (Table 1).

The specimens were cleaned of obvious surface contamination using a diamond-tipped dental burr. Then, the samples were demineralised, and the collagen was extracted following a standard modified Longin method [1-4]. Stable isotope measurements were performed in the Stable Isotope Biogeochemistry Laboratory (SIBL) at Durham University — the analytical methods are described in more detail in Gröcke *et al.* [21].

#### **Ethics Statements**

This study does not involve any modern human or animal subject.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the data reported in this article.

#### Data availability

Carbon, nitrogen and sulphur isotope data of archaeological fish and mammal bone collagen from Lithuania (Original data) (IsoArcH)

#### **CRediT Author Statement**

**Harry K. Robson:** Conceptualization, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review & editing, Visualization, Project administration; **Kurt J. Gron:** Formal analysis, Investigation, Writing – review & editing; **Darren R. Gröcke:** Formal analysis,

Investigation, Writing – review & editing; **Giedrė Piličiauskienė:** Resources, Writing – review & editing; **Gytis Piličiauskas:** Conceptualization, Resources, Writing – review & editing, Visualization

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#### References

- R. Longin, New method of collagen extraction for radiocarbon dating, Nature 230 (1971) 241–242, doi:10.1038/ 230241a0.
- [2] S.H. Ambrose, M.J. DeNiro, The isotopic ecology of east African mammals, Oecologia 69 (1986) 395–406, doi:10. 1007/BF00377062.
- [3] T.A. Brown, D.E. Nelson, J.S. Vogel, J.R. Southon, Improved collagen extraction by modified Longin method, Radiocarbon 30 (2) (1988) 171–177, doi:10.1017/S0033822200044118.
- [4] MJ. DeNiro, Postmortem preservation and alteration of in vivo bone collagen isotope ratios in relation to palaeodietary reconstruction, Nature 317 (1985) 806–809, doi:10.1038/317806a0.
- [5] K. Salesse, R. Fernandes, X. de Rochefort, J. Bružek, D. Castex, É. Dufour, IsoArcH.eu: an open-access and collaborative isotope database for bioarchaeological samples from the Graeco-Roman world and its margins, J. Archaeol. Sci. Rep. 19 (2019) 1050–1055, doi:10.1016/j.jasrep.2017.07.030.
- [6] E. Simčenka, J. Kozakaitė, G. Piličiauskienė, L. Gaižauskas, G. Piličiauskas, Human diet during the Stone Age and Early Metal period (7000–1 cal BC) in Lithuania: an update, Radiocarbon 64 (5) (2022) 1171–1189, doi:10.1017/RDC. 2022.41.
- [7] G. Piličiauskas, Virvelinės KERAMIKOS KULTŪRA LIETUVOJE 2800–2400 cal BC, Lietuvos Istorijos Institutas, Vilnius, 2018.
- [8] G. Piličiauskas, R. Jankauskas, G. Piličiauskienė, O.E. Craig, S. Charlton, The transition from foraging to farming (7000–500 cal BC) in the SE Baltic: a re-evaluation of chronological and palaeodietary evidence from human remains, J. Archaeol. Sci. Rep. 14 (2017) 530–542 a, doi:10.1016/j.jasrep.2017.06.004.
- [9] G. Piličiauskas, R. Jankauskas, G. Piličiauskienė, T Dupras, Reconstructing Subneolithic and Neolithic diets of the inhabitants of the SE Baltic coast (3100–2500 cal BC) using stable isotope analysis, Archaeol. Anthropol. Sci. 9 (7) (2017) 1421–1437, doi:10.1007/s12520-017-0463-z.
- [10] G. Piličiauskas, G. Kluczynska, D. Kisielienė, R. Skipitytė, K. Peseckas, S. Matuzevičiūtė, H. Lukešová, A. Lucquin, O.E. Craig, H.K. Robson, Fishers of the Corded Ware culture in the Eastern Baltic, Acta Archaeol 91 (1) (2020) 95–120, doi:10.1111/j.1600-0390.2020.12223.x.
- [11] G. Piličiauskas, R. Skipitytė, E. Oras, A. Lucquin, O.E. Craig, H.K. Robson, The globular amphora culture in the eastern Baltic: new discoveries, Acta Archaeol. 92 (2021) 227–251, doi:10.1163/16000390-20210037.
- [12] O. Nehlich, M.P. Richards, Establishing collagen quality criteria for sulphur isotope analysis of archaeological bone collagen, Archaeol. Anthropol. Sci. 1 (2009) 59–75, doi:10.1007/s12520-009-0003-6.
- [13] J.W. Walser III, S. Kristjánsdóttir, D.R. Gröcke, R.L. Gowland, T. Jakob, G.M. Nowell, C.J. Ottley, J. Montgomery, At the world's edge: Reconstructing diet and geographic origins in medieval Iceland using isotope and trace element analyses, Am. J. Phys. Anthropol. (2020) 142–163, doi:10.1002/ajpa.23973.
- [14] O.E. Craig, R. Ross, S.H. Andersen, N. Milner, G.N. Bailey, Focus: sulphur isotope variation in archaeological marine fauna from Northern Europe, J. Archaeol. Sci. 33 (2006) 1642–1646, doi:10.1016/j.jas.2006.05.006.
- [15] H.K. Robson, S.H. Andersen, L. Clarke, O.E. Craig, K.J. Gron, A.K.G. Jones, P. Karsten, N. Milner, T.D. Price, K. Ritchie, M. Zabilska-Kunek, C. Heron, Carbon and nitrogen stable isotope values in freshwater, brackish and marine fish bone collagen from Mesolithic and Neolithic sites in Central and Northern Europe, Environ. Arch. 21 (2) (2016) 105–118, doi:10.1179/1749631415Y.0000000014.
- [16] I. Antanaitis-Jacobs, M. Richards, L. Daugnora, R. Jankauskas, N. Ogrinc, Diet in early Lithuanian prehistory and the new stable isotope evidence, Archaeol. Balt 12 (2009) 12–30.
- [17] G.E. Fahy, C. Deter, R. Pitfield, J.J. Miszkiewicz, P. Mahoney, Bone deep: Variation in stable isotope ratios and histomorphometric measurements of bone remodelling within adult humans, J. Archaeol. Sci. 87 (2017) 10–16, doi:10.1016/j.jas.2017.09.009.
- [18] O. Nehlich, The application of sulphur isotope analyses in archaeological research: a review, Earth-Sci. Rev. 142 (2015) 1–17, doi:10.1016/j.earscirey.2014.12.002.
- [19] N.J. Van der Merwe, E. Medina, The canopy effect, carbon isotope ratios and foodwebs in amazonia, J. Archaeol. Sci. 18 (3) (1991) 249–259, doi:10.1016/0305-4403(91)90064-V.
- [20] E. Guiry, Complexities of stable carbon and nitrogen isotope biogeochemistry in ancient freshwater ecosystems: implications for the study of past subsistence and environmental change, Front. Ecol. Evol. 7 (2019) 313, doi:10. 3389/fevo.2019.00313.
- [21] D.R. Gröcke, E.R. Treasure, J.J. Lester, K.J. Gron, M.J. Church, Effects of marine biofertilisation on Celtic bean carbon, nitrogen and sulphur isotopes: implications for reconstructing past diet and farming practices, Rapid Commun. Mass Spectrom. 35 (2021) e8985, doi:10.1002/rcm.8985.