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RADIOCARBON AMS DATING OF MESOLITHIC HUMAN REMAINS FROM POLAND

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ABSTRACT. Biological studies on Mesolithic human remains from the Polish region are a rare subject of scientific research due to the limited number of these relics and their poor state of preservation. From the project titled “Old material with new methods: Using the latest bio-chemical analysis in studies of Mesolithic human remains from the Polish areas,” the radiocarbon (¹⁴C) dating of bones using accelerator mass spectrometry (AMS) has been performed. For these experiments, the gelatin was extracted from bones, and its quality evaluated by the C/N_{at} ratio and the stable isotope composition of both carbon and nitrogen. The ¹⁴C results have been obtained for 11 bone samples from 5 sites, and throughout this work the results of two preparation methods are compared. The simple gelatin extraction provided material with unsatisfactory collagen quality indicators, while additional alkali treatment allowed us to obtain much more reliable, and generally older, results. Additionally, analysis on VIRI/SIRI samples were conducted to test the developed method. Only seven of the investigated bone samples yielded ages within Mesolithic period, and the most reliable dates range from 5800 to 6800 cal BC. One sample was not datable, and two were shown to be much younger than expected.

KEYWORDS: C/N_{at}, human bones, Mesolithic, radiocarbon AMS dating, stable isotopes.

INTRODUCTION

Human remains, dating back to the Mesolithic period in Poland (8000–4500 BC), are a unique and important knowledge source of the many issues related to biology and social life in the oldest human populations (e.g. Gumiński 1995; Lillie and Richards 2000; Schulting and Richards 2001; Sulgostowska 2006). This results mainly from the limited number of well-preserved human bones, both in the whole Europe and in particular in Poland (e.g. Kozłowski 1998). Mesolithic human remains are precious and rare findings, thus their analysis was usually restricted to descriptive macroscopic research (e.g. Stęślicka-Mydlarska 1954; Szlachetko et al. 1964; Gładykowska-Rzeczycka 1973; Wiercińska and Szlachetko 1977). However, non-destructive research methods, which today are used in bioarchaeological research allow for a return to the old material (e.g. Tomczyk et al. 2014) and validation of the previously studied materials and sites, as well as reassessment of conclusions drawn dozens of years ago. At present in Europe and in the world, Mesolithic remains are a subject to detailed, multidisciplinary analyses, performed according to the latest methodological approach (e.g. Pazdur et al. 2004; Szostek et al. 2005; Meiklejohn et al. 2010). These novel technological analyses provide negligible invasion to the investigated material and does not deprive the specimens of their museum and exhibition value.

The scientific aim of the present project is to apply accelerator mass spectrometry radiocarbon (AMS ¹⁴C) dating to Mesolithic human remains (bone and sometimes tooth material) from five archaeological sites in Poland: Janisławice, Giżycko-Perkunowo, Warsaw-Grochów, Wieliszew, and Woźna Wieś. Although the discovery of Mesolithic material took place over 40 years ago, archaeological analyses only included information about the context of discovery

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of the remains, the type of burial, and artifacts associated with the graves. Similarly, anthropological research only had a macroscopic character, when the material was measured and its anatomy described (Steślicka-Mydlarska 1954; Wiercińska and Szlachetko 1977). Recent biological studies, including anthropology and genetics, were performed only for the remains of the Janisławice hunter (Stanaszek and Mańkowska-Pliszka 2013; Witas et al. 2013).

The scarcity of the analytical results for such an important archaeological material triggered this project, which is aimed at using the latest bio-chemical analysis to investigate the Mesolithic human remains from Polish regions. The project is completed collaboratively by a team of anthropologists, geneticists, physicists, and specialists in history and archaeology. The research material was transported to the Department of Biological Anthropology Cardinal Stefan Wyszyński University (Warsaw). The osteological material was described using a standard procedure as given in the *Standards for Data Collection from Human Skeletal Remains* (Buikstra and Ubelaker 1994). The protocol contains the following observations: (1) sex and age-at-death assessment, (2) metric skull measurements, (3) metric measurements of the postcranial skeleton, and (4) observation of non-metric skeletal traits. During this phase of research, the bone material was subsampled and transported to other specialist laboratories for genetic testing, isotopic determination, and ^{14}C dating.

The results presented hereafter will focus on the ^{14}C analysis of the remains by AMS, stable carbon ($\delta^{13}\text{C}$) and nitrogen ($\delta^{15}\text{N}$) isotope determination, as well as the C/N ratio; which will be mainly used as sample (in fact, the “collagen” *sensu* van Klinken 1999) quality indicators. The oxygen isotope composition, sequencing of genetic material, and the analysis of changes in the tooth enamel using SEM and x-ray techniques is being conducted simultaneously by collaborative research teams. Moreover, the extensive isotopic studies on the diet reconstruction, including measurement of the components in the food chain is an ongoing study.

The diagnostics of collagen extracted from bones for the purpose of ^{14}C dating and isotope studies can be provided by several measurable parameters (DeNiro 1985; Ambrose 1990; van Klinken 1999). The collagen yield should exceed 1%, and the percentage of elements should be 15.3–47% for carbon and 5.5–17.3% nitrogen, by mass. Atomic $\text{C}/\text{N}_{\text{at}}$ ratios, indicating good preservation, should ideally fall within the range equal to the values obtained for modern animals and humans ($\text{C}/\text{N}_{\text{at}} = 2.9\text{--}3.6$; Ambrose 1990). Following the work of Brock et al. (2010) and Tisnerat-Laborde et al. (2003), a $\text{C}/\text{N}_{\text{at}}$ higher than recommended values indicates on a strong diagenesis or/and the presence of considerable amount of humic substances.

The carbon and nitrogen stable isotopic ratios of collagen depend on the diet of an individual. In a typical European inland case, exclusive of C_4 plants and marine food resources, the $\delta^{13}\text{C}$ value for adult bone collagen falls within a range of -19‰ to -22‰ , while $\delta^{15}\text{N}$ may cover a much wider range, e.g. from 2‰ to 12‰ (van Klinken 1999). For the Eastern Baltic area (inland areas in Lithuania), the values for the Late Mesolithic and Subneolithic bones range from -21‰ to -23.5‰ for $\delta^{13}\text{C}$ and 10.5‰ to 13‰ for $\delta^{15}\text{N}$ (Piličiauskas et al. 2018).

Elevated $\delta^{15}\text{N}$ values could indicate a considerable proportion of marine or freshwater component in the diet, which should imply the application of a relevant reservoir correction to the ^{14}C dates (e.g. Schulting and Richards 2001; Cook et al. 2001; Olsen et al. 2010; Svyatko et al. 2015; Marchenko et al. 2015). However, for the SE Baltic region the freshwater reservoir effect (FRE) reported by Piličiauskas and Heron (2015) is highly variable and site dependent, extending from non-present to a few centuries.

In case of children, breast-feeding alters the stable isotope composition. In particular, the $\delta^{15}\text{N}$ increases by ca. +3‰ during breast-feeding, and after weaning the $\delta^{15}\text{N}$ value approaches that of adults with a similar diet (e.g. Fuller et al. 2006). Also, collagen $\delta^{13}\text{C}$ values in young children's bones are enriched, due to the so-called "carnivore" effect, which may reach ca. 1‰ (e.g. Richards et al. 2002; Fuller et al. 2006).

In any case, a combination of all $\text{C}/\text{N}_{\text{at}}$ and stable isotope data measured on exactly the same material, which was subjected to ^{14}C dating, provides a valuable tool for either pre-screening the material before dating or critical evaluation of previously obtained ^{14}C dates, as shown by e.g. van Klinken (1999), Svyatko et al. (2015), and Marchenko et al. (2015). Scirè Clabrisotto et al. (2013) reported that reasonable agreement with expected age may be obtained when $\text{C}/\text{N}_{\text{at}}$ is slightly above the upper limit of the recommended range.

MATERIAL

This manuscript reviews the osteological material from five Mesolithic sites in Poland. In addition, three bone samples from international ^{14}C intercomparison programmes VIRI and SIRI (Scott et al. 2010, 2017) were subjected to the same ^{14}C dating procedures as archaeological samples.

The sites are located in two regions in Poland: Mazovia (Janisławice, Warsaw-Grochów, Wieliszew XI) and northeastern Poland (Giżycko-Pierkunowo, Woźna Wieś). The location of the sites is presented in Figure 1. The material was deposited at the State Archaeological Museum in Warsaw and the Institute of Archaeology and Ethnology of the Polish Academy of Sciences in Warsaw.

Janisławice

The most famous Mesolithic skeleton from Poland comes from Janisławice (51°50'44"N 20° 03'18"E; Chmielewska 1954; Figure 1). These are the human bone remains of a 30 to 40-year-old male and were discovered in a sitting position, the find was excavated in 1936–1937. The remains were accompanied by numerous artifacts identified as the hunter's accessories. The remains were initially investigated and described in detail by Stęślicka-Mydlarska (1954) and these observations were revised a couple of times later (Cyrek 1978; Sulgostowska 1990a; Stanaszek and Mańkowska-Pliszka 2013). The first ^{14}C analysis of the red deer antler was conducted in 1975 but was unsuccessful due to an insufficient collagen amount (Sulgostowska 1990a). The ^{14}C dating of collagen from the femur bone was performed in the Gliwice Radiocarbon Laboratory in 1985 with use of the radiometric technique (gas proportional counting) and provided an age of 6580 ± 80 ^{14}C BP (Gd-2432), as reported by Sulgostowska (1990a) and Pazdur et al. (1994). The anthropological studies were carried out in 2015, but they did not contain any chronological analysis (Stanaszek and Mańkowska-Pliszka 2015). From this skeleton, two fragments were subjected to this present study: one from femur (named Janisławice Femur) and the second from tibia bone (Janisławice Tibia). The bones have not been subjected to any conservatives.

Additional bone fragments from the Janisławice hunter were discovered in a museum collection archive. A sample of cortical bone fragment was acquired for ^{14}C AMS dating (named Janisławice 2), however, the placement and description of this finding was unsatisfactory, thus the association of this material with the previously described hunter bones is uncertain.



Figure 1 Location of the five Mesolithic sites from the two regions: northeastern Poland (Giżycko-Pierkunowo, Woźna Wieś) and the Mazovia region (Wieliszew, Warsaw-Grochów, Janisławice). The insets present the osteological material collected at each site. Background map from www.google.com/maps.

Giżycko-Pierkunowo

In July 1965, two graves with skeletons were explored in the Pierkunowo village, near Giżycko in the Mazurian Lakeland (54°04'20"N 21°43'50"E; Figure 1) by the State Archaeological Museum in Warsaw. The graves were situated about 35m from the south-eastern shore of Lake Kisajno. The graves were flat pit-graves without any stone setting. The skeletons, found in lying position, were dyed with ochre (Głosik 1969a).

Four skeletons from three separate graves were sampled for this study. Samples Giżycko 1 (rib fragment from ca. 3-yr-old child skeleton) and Giżycko 4 (phalanx of female adult, 35–39 yr old), both come from the first grave. From the second grave, phalanx bones of a child, ca. 18 months old, were collected (sample named Giżycko 2), and rib bones of an adult (sex and age undetermined) came from the third grave and termed Giżycko 3. The material was not conserved.

Prior to the ^{14}C dating, a chemical method based on fluorine and chlorine content in bone mineral fraction (Wysoczański-Minkowicz 1979) was applied to the female adult bone from this site. The obtained age was 3750 ± 150 BC (Głosik 1969b), which is inconsistent with the archaeological evidence.

Warsaw-Grochów

The human remains (only the cranium, see Figure 1) of so-called “little girl from Grochów” (8–9 yr old) were accidentally discovered in 1961 and they are considered to be the oldest

excavated human bones in the Warsaw area. The discovery took place during trench digging on the Nowokinowa street in Grochów (eastern part of Warsaw; 52°14'N 21°1'E). The remains have been found in a layer deposited over the Vistula River, and by stratigraphical relative dating it was assumed, that the skull was about 7000 yr old. However, the remains were not accompanied by any archaeological material (Szlachetko et al. 1964) and no chemical analysis has been conducted so far. The skull may have been subjected to conservation.

Wieliszew

In 1961, the human remains from Wieliszew XI were excavated from a large single dune situated on the left bank of the River Narew (52°27'00"N 20°58'08"E). The site is approximately 1 km from the river (Więckowska 1985). The human material consists of numerous small bone fragments (Figure 1) with a light yellowish color and some gray infusions. A strong degree of mineralization together with cracks of the lamina suggest that this material was cremated. However, the lack of significant deformation of these particular bone fragments shows that the material was not subjected to high temperatures (Wiercińska and Szlachetko 1977). Thus, cremation at low temperature is proposed. In 1962, three fragments of these bones were investigated by using the fluorine-apatite (F/Apatite) method, giving an estimated date of about 4900–4100 BC, but further revision by Wysoczański-Minkowicz (1979) with the use of the fluoro-chloro-apatite method (F/Cl/Apatite) provided an older age of ca. 5850 BC.

Woźna Wieś

The fragments of a human cranium belonging to an adult with an undetermined age and gender were discovered in 1961 in Woźna Wieś, a village near the Dręstwo Lake, from which the River Jędrzonia outflows, belonging to the Elk Lakeland (53°40'53"N 22°45'06"E; Sulgustowska 1990b; Tobolski and Żurek 2012). Paleolithic, Mesolithic, and Neolithic settlements have been discovered on a sandur (outwash plain) and excavated over the years 1974–1978. The traces of the settlement occurred in the lakeside arable fields approximately 500 m from the exiting River Jędrzonia. In addition to the abundant flint artifacts, moose and reindeer remains were found in a layer dated to the Allerød interstadial, as well as subsequent forest animal remains (bison, deer, sheep, and horse) and human bones. The fragments have been glued together to reconstruct the skull shape (see Figure 1).

METHODS

The research methods applied within this study comprise firstly of collagen extraction from bones according to a modified Longin's protocol in the Gliwice Radiocarbon Laboratory (Piotrowska and Goslar 2002; Piotrowska 2013). All bone samples were cleaned in an ultrasonic bath in demineralized water, then dried and ground in a ball mill. The powdered bone was treated with use of 0.5M hydrochloric acid in a glass vial at a room temperature to decompose the mineral fraction. The acid was replaced several times, and the reaction was considered complete when pH stabilized at <1 and no bubbles were observed. The whole procedure took 1–2 working days. The insoluble residue was rinsed with demineralized water to neutral pH. Next, gelatinization was performed for all the samples: the residue was acidified and kept in 80°C for 12 hr in an acidic solution (HCl, pH = 3). The obtained supernatant was centrifuged, filtered, put in a glass vial and dried in an oven at 75°C. Ultrafiltration was not used. Hereafter, we refer to recovered material as the gelatin, and to this treatment as Treatment A.

In Treatment A, the demineralized residue was not subjected to any NaOH treatment, due to the risk of collagen loss. The second batch of samples was treated with 0.1M NaOH for 30 min at a room temperature, after the demineralization step, and rinsed with demineralized water to a neutral pH. Next, the gelatinization was performed as described above. Hereafter, we refer to this procedure as Treatment B.

The subsample of gelatin was subjected to graphite preparation using an AGE-3 system equipped with a VarioMicroCube by Elementar elemental analyzer and automated graphitization unit (Nemec et al. 2010; Wacker et al. 2010). This analyzer (hereafter called VMC-EA) was calibrated with use of acetanilide and sulphanilamide reference materials to obtain the %C, %N and C/N atomic ratios. The ^{14}C concentrations in graphite produced from unknown samples, Oxalic Acid II standards, and coal blanks have been measured by the DirectAMS laboratory, Bothell, USA (Zoppi et al. 2007; Zoppi 2010). The results are reported in Table 1 (reference samples) and Table 2 (archaeological samples). The ^{14}C dates have been subjected to calibration with the use of OxCal v4.3.2 (Bronk Ramsey 2009) and IntCal13 calibration curve (Reimer et al. 2013).

Another gelatin subsample was assigned for stable isotope analysis of the carbon and nitrogen ($\delta^{13}\text{C}$, $\delta^{15}\text{N}$), %C, %N, and C/N_{at} quantities by using a CF-EA-IRMS system working in the Gliwice Mass Spectrometry Laboratory. The equipment comprises of a EuroVector elemental analyzer and continuous-flow IsoPrime mass spectrometer. The instrument precision is 0.1‰ for $\delta^{13}\text{C}$ and 0.3‰ for $\delta^{15}\text{N}$. The elemental analyzer (hereafter called EV-EA) was calibrated for C/N_{at} ratios with use of UREA and EMA P2 standards. At least three subsamples from each collagen sample have been prepared, along with the standards: IAEA-C8, IAEA-C5 and EMA P2 for carbon, as well as NO₃ and USGS34 for nitrogen. The reported values of $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ have been normalized to the VPDB and AIR scales, respectively. The average values are calculated and presented in Table 2 and in Figure 4.

The two elemental analyzers require considerably different sample masses. One gelatin subsample of 3–5 mg was combusted for graphitization in VMC-EA, while for EV-EA the required sample masses of approximately 0.25–0.30 mg.

Two of the reference bone samples (VIRI H and E, GdA-5341 and 5342) were prepared in the second batch of samples. The SIRI C (GdA-5339) bone was prepared in 2014 with the inclusion

Table 1 Results for international ^{14}C intercomparison bone samples, prepared with Treatment B method (gelatinization and NaOH wash). Consensus values after Scott et al. (2010) for VIRI and Scott et al. (2017) for SIRI. LoB: limit of blank, i.e. the highest apparent ^{14}C concentration reported as F_m value (no correction for background).

Lab no. GdA-	Sample	Coll. yield (%)	%C	%N	C/N _{at} VMC-EA	Consensus value	Age ^{14}C BP
5341	VIRI H Whale bone	13	37.3	13.5	3.2	9528 ± 7	9535 ± 45
5342	VIRI E Mammoth bone	11	41.3	14.9	3.2	39305 ± 21	35600 ± 480
5339	SIRI C Mammoth bone (collagen)	—	28.1	10.6	3.1	0.00895 (LoB)	$F_m = 0.00679$ ± 0.00020

Table 2 Results of C/N_{at}, stable isotope, ¹⁴C determinations, and calibration. Treatment method A: gelatinization, treatment method B: gelatinization with alkali wash. Elemental analyzers: EV—EuroVector (connected to IRMS system), VMC—VarioMicroCube (connected to graphitization system). The calibration was performed with the use of OxCal v4.3.2 (Bronk Ramsey 2009) and IntCal13 calibration curve (Reimer et al. 2013). * = radiocarbon date reported by Sulgostowska (1990a), recalibrated.

Sample name	Treat. meth.	Lab no. GdA-	Coll. yield, %	EA used	%C	%N	C/N _{at}	δ ¹³ C, ‰ VPDB	δ ¹⁵ N, ‰ AIR	Age ¹⁴ C BP	Calibrated age range (95.4%)
Janisławice Tibia <i>Cortical bone fragment</i>	A	4236	4.3	EV	12.1	4.0	3.5	-22.2 ± 0.1	10.4 ± 0.1	5875 ± 40	95.4% probability 4840BC (93.3%) 4670BC 4635 BC (2.1%) 4620BC
Janisławice Femur <i>Cortical bone fragment</i>	A	4237	—	EV	23.8	7.1	3.9	-21.5 ± 0.1	11.2 ± 0.1	6570 ± 40	95.4% probability 5615BC (11.8%) 5590BC 5570BC (83.6%) 5475BC
Janisławice 2 <i>Cortical bone fragment</i>	B	5133	5.9	EV	39.5	13.3	3.5	-20.7 ± 0.2	10.7 ± 0.1	6885 ± 30	95.4% probability 5840BC (95.4%) 5715BC
				VMC	44.3	15.0	3.4				
Janisławice 2 <i>Cortical bone fragment</i>	B	5288	5.0	EV	41.6	15.1	3.2	-20.7 ± 0.2	9.8 ± 0.1	4940 ± 60	95.4% probability 3940BC (10.6%) 3860BC 3815BC (84.8%) 3635BC
				VMC	44.6	15.9	3.3				
Janisławice Jan.1* <i>Femur bone (complete)</i>	A	Gd-2432	—	—	—	—	—	—	—	6580 ± 80	95.4% probability 5645BC (95.4%) 5375BC
Giżycko 1 <i>Rib fragment, 3-yr-old child</i>	A	4562	11.4	EV	4.27	0.98	5.1	-20.3 ± 0.1	13.8 ± 0.3	7050 ± 35	95.4% probability 6010BC (93.7%) 5875BC 5860BC (1.7%) 5850BC
	B	5134	8.5	EV	37.7	13.3	3.3	-19.4 ± 0.1	13.8 ± 0.2	7770 ± 35	95.4% probability 6655BC (95.4%) 6500BC
Giżycko 4 <i>Phalanx, female adult</i>	A	4565	22.3	EV	1.90	0.33	6.7	-22.4 ± 0.1	10.0 ± 0.1	6200 ± 25	95.4% probability 5280BC (0.5%) 5275BC 5225BC (94.9%) 5055BC
	B	5131	2.7	EV	36.9	13.2	3.3	-20.6 ± 0.1	11.9 ± 0.2	7600 ± 45	95.4% probability 6565BC (2.8%) 6545BC 6530BC (92.6%) 6390BC
				VMC	38.7	13.1	3.4				

Table 2 (Continued)

Sample name	Treat. meth.	Lab no. GdA-	Coll. yield, %	EA used	%C	%N	C/N _{at}	δ ¹³ C, ‰ VPDB	δ ¹⁵ N, ‰ AIR	Age ¹⁴ C BP	Calibrated age range (95.4%)
Giżycko 2 <i>Phalanx, 1.5-yr-old child</i>	A	4563	8.9	EV	4.28	0.96	5.2	-20.9 ± 0.2	13.8 ± 0.4	7275 ± 35	95.4% probability 6220BC (95.4%) 6065BC
Giżycko 3 <i>Rib, adult</i>	A	4564	8.6	EV	7.1	1.9	4.4	-19.8 ± 0.1	13.8 ± 0.2	7230 ± 30	95.4% probability 6210BC (28.3%) 6135BC 6125BC (67.1%) 6025BC
Warsaw Grochów <i>Cranium, 9-yr-old female child</i>	B	5130	11.3	EV VMC	39.0 43.6	13.7 15.3	3.3 3.3	-19.6 ± 0.1	14.5 ± 0.1	7860 ± 30	95.4% probability 6815BC (95.4%) 6635BC
	A	4566	26.9	EV	22.7	6.3	4.2	-15.9 ± 0.2	8.7 ± 0.1	2820 ± 25	95.4% probability 1040BC (95.4%) 910BC
Wieliszew <i>Small bone fragments</i>	B	5132	12.5	EV VMC	39.2 46.1	13.1 15.4	3.5 3.5	-16.6 ± 0.1	10.5 ± 0.2	3075 ± 30	95.4% probability 1415BC (95.4%) 1260BC
	A	4568	14.9	EV	0.093	0.022	4.9	-23.8 ± 0.1	5.7 ± 0.4	—	—
Woźna Wieś <i>Cranium, adult</i>	B	5129	1.6	EV	0.43	0.15	3.3	—	—	—	—
	A	4567	20.0	EV	0.23	0.026	10.3	-25.0 ± 0.3	5.4 ± 0.1	510 ± 40	95.4% probability 1320AD (14.7%) 1355AD 1390AD (80.7%) 1450AD
	B	5135	4.5	EV VMC	36.5 38.1	12.8 12.5	3.3 3.5	-19.4 ± 0.1	13.7 ± 0.2	195 ± 40	95.4% probability 1640AD (23.3%) 1700AD 1720AD (47.9%) 1820AD 1830AD (5.6%) 1880AD 1915AD (18.6%) ...

of alkali treatment (Piotrowska and Goslar 2002) and the stored gelatin was re-dissolved, filtered with a nylon woven net filter, and dried before combustion and graphitization.

RESULTS AND DISCUSSION

The measurement results for reference bone samples are listed in Table 1, and the results of the archaeological samples for both treatments are listed in Table 2. Unfortunately, the material for two of the samples (Giżycko 2 and Janisławice Tibia S.) was not available for repeated analysis with alkali treatment. The calibration plots are presented in Figure 3, while Figure 4 shows the stable isotope data.

Quality Indicators and ¹⁴C Results

The first batch of gelatin, prepared without a NaOH wash by Treatment A, yielded unsatisfactory results, in terms of the C/N_{at} ratios (3.5–10.3) and depleted %C and %N for almost all the samples (Table 2). The average gelatin yield was 14.6%. Therefore, additional sample material was collected, and the preparation procedure was repeated with an alkali treatment step (Treatment B).

Most of the Treatment B samples yielded less gelatin (6.5% on average) than the previous ones. Quality indicators fell within acceptable ranges, with C/N_{at} values ranging from 3.2 to 3.5 and the expected carbon and nitrogen content (Table 2). In addition, two independent analysis were performed with two elemental analyzers. The results for C/N_{at} values do not differ by more than 0.2. This difference is concordant with the reported values for laboratory inter-comparison studies (Sealy et al. 2014), and the uncertainty of C/N results reported by Scire-Clabrisotto et al. (2013) and Svyatko et al. (2015).

The results for reference bone material indicate a good quality of gelatin with C/N_{at} 3.1–3.2 (Table 1). The VIRI H determined age agrees perfectly with the consensus value within 1-sigma interval. The SIRI C sample, which may be regarded as background material, gave a F_m value 0.00679 ± 0.00020 , which is even lower than the reported consensus value. The most confusing result was obtained for VIRI E, which gave an age ca. 3700 ¹⁴C yr younger than consensus value. While it is a considerable shift, a very wide range of results for this particular bone sample was reported by Scott et al. (2010): the interquartile values were 35,320 and 40,400 ¹⁴C BP (n = 57). After the outliers were omitted the interquartile results, from AMS, were 36,540 and 40,648 ¹⁴C BP (n = 40). The resulting consensus value was calculated to $39,305 \pm 121$ ¹⁴C BP, based on n = 28 dates. According to the methodology applied by Scott et al. (2010), our result would not be considered as an outlier removed in the first step. The difference from the median (equal to 39,695 ¹⁴C BP) is 4095, which is less than 7620 (1.5 times the interquartile range, IQR = 5080). However, our result is on the edge of acceptability and indicates an action should be undertaken to further evaluate the reasoning behind this data. It is most likely that our method was not sufficient enough to extract the material with a proper purity. The use of longer alkali treatment, ultrafiltration or a selection of more specific chemical components should be considered. Despite the disputable result for the VIRI E sample, which proved to be problematic for many laboratories, the demonstrated reasonable ¹⁴C background level allows to expect our results to be accurate.

Five of the ¹⁴C ages obtained for the archaeological samples from Treatment B are older than the ¹⁴C ages for Treatment A by 250–1400 ¹⁴C yr. This indicates the presence of a

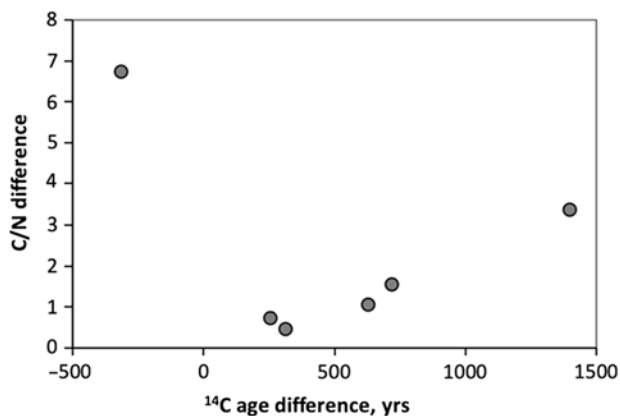


Figure 2 Differences in the C/N_{at} ratios and the ^{14}C ages between the results obtained for Treatment A (simple gelatinization) and Treatment B (gelatinization with alkali wash).

contaminant which is younger than the age of bones. Adding this to elevated C/N_{at} ratios for the Treatment A gelatin, the most probable contaminant is a substance rich in carbon, which was removed by the alkali treatment in Treatment B. The difference between ages for the same samples subjected to Treatments A and B is proportional to the difference in C/N_{at} ratios for the same samples (Figure 2). Also, the $\delta^{13}\text{C}$ was shifted towards values more common for human bone collagen: increased for four of them (Gizycko and Janisławice sites) and decreased for Warsaw-Grochów sample (Figure 3).

In the case of the Woźna Wieś sample, the new date is 320 yr younger. This sample is also characterized by a significant C/N_{at} shift from 10.3 to 3.5 between the Treatments A and B, and a huge difference in %C and %N. In this case the contaminant had a carbon content much higher than collagen, and was older than the bone sample. The significant improvement in a quality of dated material is seen also in $\delta^{13}\text{C}$ (shift from -25‰ to -19.4‰) and $\delta^{15}\text{N}$ (from 5.4‰ to 13.7‰).

Janisławice

For the Janisławice hunter, two cortical bone fragments have been subjected to ^{14}C AMS dating with Treatment A. The one from the tibia bone yielded an age of 5875 ± 40 BP ($C/N_{\text{at}} = 3.5$), while the femur bone gave a distinctly older result by ca. 700 yr (6570 ± 40 BP, $C/N_{\text{at}} = 3.9$). This discrepancy is too large to be explained by turnover time in different bone fragments from a single individual, which is dozens of years higher. In the Treatment B the material was only available for the femur bone, and yielded a lower C/N_{at} ratio (3.4–3.5), an acceptable %C and %N, and gave an even older age of 6885 ± 30 BP.

For the Treatment A the values of $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ are higher for the femur bone (-21.5‰ , 11.2‰) than for the tibia (-22.2‰ , 10.4‰). The $\delta^{13}\text{C}$ is even higher for the femur sample treated with alkali (-20.7‰). The $\delta^{13}\text{C}$ shows a rising tendency along with the decreasing C/N_{at} . The humic substances, which are suspected to alter the Treatment A results, would most probably have a $\delta^{13}\text{C}$ lower than -25‰ and high C/N_{at} ratio. Therefore, the observed $\delta^{13}\text{C}$ and C/N_{at} trends confirm this hypothesis.

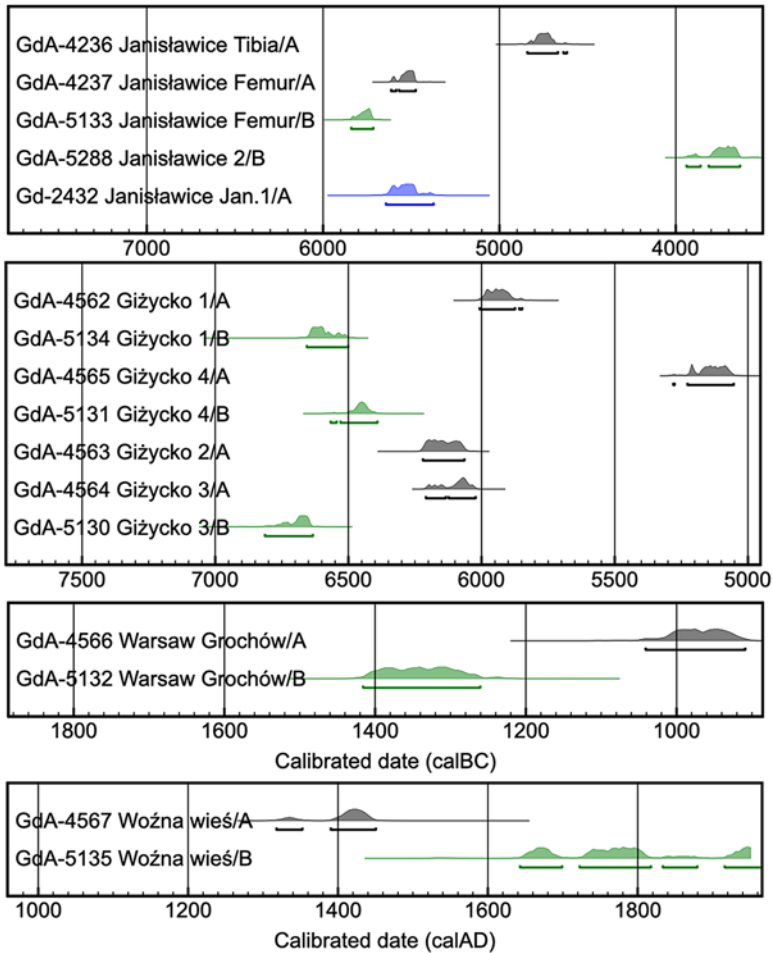


Figure 3 The calibration results of the ¹⁴C dates; gray: Treatment A (simple gelatinization), green: Treatment B (gelatinization with alkali wash), blue: radiometric date. The ¹⁴C dates have been subjected to calibration with the use of OxCal v4.3.2 (Bronk Ramsey 2009) and IntCal13 calibration curve (Reimer et al. 2013). (Please see electronic version for color figures.)

An AMS age of 6570 ± 40 BP (GdA-4237) is in perfect accordance with the result of 6580 ± 80 (Gd-2432) obtained by radiometric ¹⁴C dating on another femur bone (see Figure 3). Both samples were prepared with a similar methodology, namely without alkali treatment, and a similar influence of humic substances on the ¹⁴C age can be deduced.

The youngest age 4940 ± 60 BP was obtained for the newly acquired bone fragment. The quality of the gelatin is acceptable with a $C/N_{at} = 3.3$ (44.6% C and 15.9% N), and stable isotope composition of $\delta^{13}C = -20.7\text{‰}$, in agreement with Janisławice femur bone. The $\delta^{15}N = 9.8\text{‰}$ is, however, lower by almost 1‰ in comparison with femur bone (see Figure 3). Thus, the discrepancy in the age with other Janisławice samples cannot be explained by contamination, but rather shows it is a fragment of another skeleton, younger by almost 2000 yr. Given the dubious provenience of this sample, this age should be regarded as unreliably associated with the Janisławice hunter skeleton.

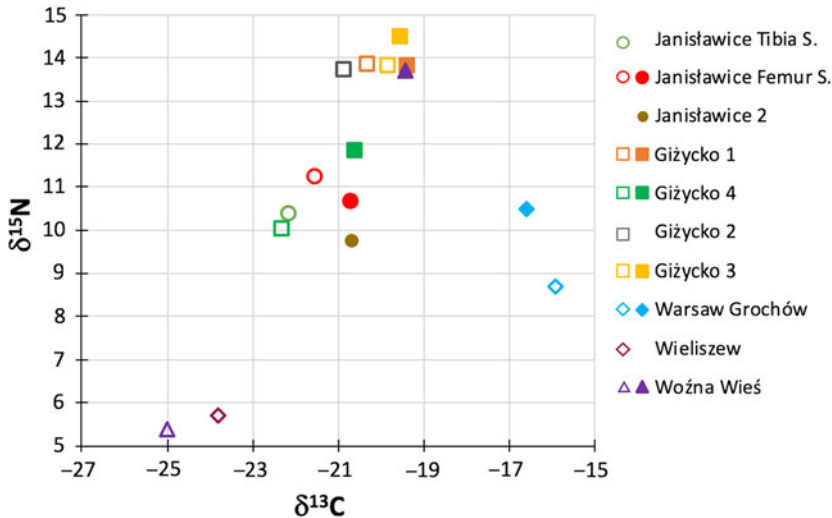


Figure 4 The stable isotope composition of investigated samples. Open symbols: Treatment A (simple gelatinization), filled symbols: Treatment B (gelatinization with alkali wash).

In the light of the obtained results the most reliable age of Janisławice hunter skeleton is connected with sample from femur bone, prepared with alkali wash, which is 6885 ± 30 BP, and 5840–5715 cal BC (Table 2, Figure 3).

Giżycko-Pierkunowo

Four bone samples from this site have been subjected to analyses. All the C/N_{at} values for the samples prepared with Treatment A were higher than for collagen (ranging from 4.4 to 6.8), but still the ages fell within the Mesolithic period. The gelatin samples from the Treatment B are characterized by a considerably lower C/N_{at} ratios (3.3–3.5) and their ^{14}C ages are shifted towards older values by 630, 720, and 1400 yr. The age difference is proportional to the C/N_{at} shift (Figure 2), implying the presence of the same contaminant, a younger material of elevated C/N_{at} , which was removed during the alkali treatment.

The stable carbon and nitrogen isotope composition of the Giżycko samples is also altered by alkali treatment, most noticeably for the sample Giżycko 4, along with the highest C/N_{at} and ^{14}C age shift (Figure 4). In all cases the $\delta^{13}C$ results for Treatment B are shifted towards more positive values. Therefore, the contamination by the humic substances of high carbon content and $\delta^{13}C$ around -25‰ is the most probable explanation for the rejuvenated ages obtained by Treatment A.

The additional material was not available for the Giżycko 2 sample, which in the first trial (Treatment A) gave gelatin with a $C/N_{at} = 4.9$ and age of 7275 ± 35 BP. Following the trend presented in Figure 2, the correct age of this sample can be estimated to be at least 600–700 yr older.

Freshwater food consumption by humans from Giżycko-Pierkunowo site is likely, as the location of the site is in a close vicinity to Lake Kisajno. Therefore, the freshwater reservoir effect (FRE), causing the ages to appear older than the actual age, is likely.

The FRE is a strictly local effect, dependent on the reservoir age of lake-derived food and the proportion of this food in an individual's diet. According to the data presented by Sensuła et al. (2006), for Polish lakes the reservoir age for the organic fraction of lake sediments were: $T_R = 171 \pm 76$ ¹⁴C yr for Lake Wigry (NE Poland, 90 km E from lake Kisajno), $T_R = 539 \pm 60$ ¹⁴C years for Lake Gościąg (central Poland, 230 km SW), and $T_R = 500 \pm 200$ ¹⁴C yr for Lake Samle Duże (NE Poland, 85 km E). Using any of these values as FRE estimate may lead to incorrect conclusions, as the organic fraction of lake sediment undoubtedly contain some amount of terrestrial plants. Thus, the presence of a T_R reaching a few centuries for Lake Kisajno is to be expected and regarded as minimal FRE estimate.

The stable carbon and nitrogen isotope composition of human bones is also affected by freshwater food consumption. The $\delta^{13}\text{C}$ of the possible aquatic dietary component and the aquatic plants in NE Poland, are characterized by having relatively low $\delta^{13}\text{C}$ values, close to terrestrial C_3 plants. For the Wigry Lake, the average $\delta^{13}\text{C}$ for contemporary aquatic plants is ca. -25‰ , while for terrestrial C_3 plants the $\delta^{13}\text{C}$ is equal to ca. -26‰ . For the Gościąg Lake, the values are ca. 2‰ lower (Sensuła et al. 2006). Similarly, Reitsema (2012) has shown a $\delta^{13}\text{C}$ for fish bones ranging from -21.6‰ to -28.2‰ . Consequently, the freshwater fish consumption may not be distinguishable by the $\delta^{13}\text{C}$ values.

For fish from Polish lakes the relatively wide range of $\delta^{15}\text{N}$ was reported by Reitsema (2012): 6.6‰ to 12.1‰ for medieval samples from Central Poland. The $\delta^{15}\text{N}$ for two investigated bones, Giżycko 1 (3-yr-old child): 13.8‰ , and Giżycko 3 (adult): 14.5‰ , are even more positive. For the adult it indicates a greater proportion of freshwater fish in the diet. Conversely, a $\delta^{15}\text{N} = 11.9\text{‰}$ for another female adult (Giżycko 4) does not suggest a freshwater diet component.

The $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ of the 3-yr-old child's bones (Giżycko 1) are shifted by ca. $+2.6$ and $+1.2\text{‰}$, respectively, when compared with the values obtained for a female from the same grave (Giżycko 4). These differences are within the range expected for breast-feeding effect, or, less probable, are an effect of the elevated proportion of freshwater protein in a child's diet. The ¹⁴C ages of the two samples are disparate within 2-sigma: 7770 ± 35 ¹⁴C BP (child) and 7600 ± 45 ¹⁴C BP (female), but the calibrated age ranges overlap in the period 6570–6500 cal BC. Therefore, a coeval burial of a mother and her child cannot be excluded.

In order to estimate the FRE quantitatively, paired ¹⁴C dating of terrestrial vs. freshwater species is typically carried out (e.g. Cook et al. 2001; Olsen et al. 2010; Piličiauskas and Heron 2015; Svyatko et al. 2015), which was unavailable for this site. An extensive research into stable isotope for a particular site is required and then the application of thorough modelling tools allows one to calculate the proportion of freshwater to terrestrial component in the diet (e.g. Sayle et al. 2016). A more detailed study on the isotope-based diet reconstructions may provide a prospect to re-evaluate the dates from the Giżycko site, until then the dates should be regarded as maximal.

Warsaw-Grochów

The skull of the 8/9-yr-old girl accidentally excavated in the riverine sediments, in Warsaw, was dated to an age a much younger age than expected stratigraphy. Although the carbon and nitrogen content were satisfactory (22.7% C, and 6.3% N), the $\text{C}/\text{N}_{\text{at}} = 4.2$ ratio of the first batch of gelatin was higher than the modern values, and the stable isotope results were as unexpected ($\delta^{13}\text{C} = -15.9\text{‰}$ and $\delta^{15}\text{N} = 8.7\text{‰}$). Therefore, the preservative(s) which may have been used are the suspected cause of the discrepancy in the results. Repeated

preparation provided material which had good quality indicators (12% collagen yield, 46% C, 15% N, $C/N_{at} = 3.5$), and the age was shifted by almost 300 years to 3075 ± 30 ^{14}C BP (1415–1260 cal BC). Still, the result does not confirm the Mesolithic age of this bone.

The stable isotope composition results are $\delta^{13}\text{C} = -16.6\text{‰}$ and $\delta^{15}\text{N} = 10.5\text{‰}$. The elevated $\delta^{13}\text{C}$ indicates that the girl's diet was enriched in the ^{13}C isotope. At the present state of study, it might be cautiously concluded, that the girl consumed some C_4 plant, e.g. millet, which has a carbon isotopic signature of ca. $\delta^{13}\text{C} = -11\text{‰}$ (e.g. An et al. 2015) and which was been cultivated and consumed in Poland since ca. 2000 BC (Hunt et al. 2008; Reitsema 2012).

Wieliszew

The bones sample from the Wieliszew site yielded extremely small amount of gelatin residue, which was sufficient enough to only perform IRMS and C/N_{at} measurements, requiring micrograms of carbon, and attempts to obtain a graphite were unsuccessful. The C/N_{at} ratio of this material, from the Treatment A, was 4.9, clearly indicating the presence of a non-collagen component. Also, the $\delta^{13}\text{C} = -23.8\text{‰}$ and $\delta^{15}\text{N} = 5.7\text{‰}$ were unusual for a human bone, when compared to other results from this study. The second batch sample gave a collagen yield of 1.5% and a $C/N_{at} = 3.3$, which are acceptable values, but the carbon and nitrogen content was far too low (0.43% and 0.15%). No other results were achievable due to the low amount of recovered material.

As described by Wysoczański-Minkowicz (1979), the bones have been burned but not subjected to high temperatures (Wiercińska and Szlachetko 1977), and in this process the collagen must have almost been completely lost. Wysoczański-Minkowicz (1979) attempted to date the bones with use of fluoro-chloro-apatite method (F/Cl/Apatite) and the result provided an age of ca. 5850 BC, which agrees with archaeological evidence and remains the only available independent age determination for this material.

Woźna Wieś

The first ^{14}C result obtained for Woźna Wieś sample using the Treatment A (GdA-4567, 510 ± 40 ^{14}C BP) gave unexpectedly young results, which was inconsistent with archaeological evidence. The quality indicators prove the unreliability of the dating result, namely the carbon and nitrogen content were extremely low, 0.23 and 0.026%, respectively. The C/N_{at} ratio was equal to 10.3, while $\delta^{13}\text{C} = -25\text{‰}$ and $\delta^{15}\text{N} = 5.4\text{‰}$. The second date (Treatment B, GdA-5135) was obtained on material with quality indicators characteristic of collagen ($\%C = 37$, $\%N = 13$, $C/N_{at} = 3.5$, $\delta^{13}\text{C} = -19.4\text{‰}$ and $\delta^{15}\text{N} = 13.7\text{‰}$), but gave an even younger age 195 ± 40 ^{14}C BP. We conclude, that the dated material was contaminated by a component much older than the actual age of this sample. The glue, which had been used to reconstruct the skull, must have been present in the material despite physical cleaning before preparation, but the alkali treatment was sufficient enough to remove it. In any case, the dating results do not confirm a Mesolithic age for this material.

CONCLUSIONS

The Mesolithic age was confirmed for the Janisławice hunter, which can be placed at 5840–5715 cal BC. Similarly, the Giżycko-Pierkunowo site ages fall within the Mesolithic period. The youngest bone was determined to be 6570–6390 cal BC for the female adult

(Giżycko 4), and two other bones Giżycko 1 (child, ca. 3 yr old) and Giżycko 3 (adult, sex and age undetermined) gave slightly overlapping age ranges of 6660–6500 and 6815–6635 cal BC, respectively. However, their $\delta^{15}\text{N}$ indicated a proportion of freshwater fish in the diet, which can bias the ¹⁴C dating results. The FRE for this site may reach a few centuries, thus their true ages may fall closer to Giżycko 1 in age.

The skull of the “little girl from Grochów” was dated to 1415–1260 cal BC, thus it is not of Mesolithic age. Similarly, the bone from Woźna Wieś was dated to a much younger age, 1640–1880 cal AD. The sample from Wieliszew yielded no datable collagen material.

The investigated bone samples have been difficult material for ¹⁴C dating. The presented research confirms, that ¹⁴C dating of such relics should be accompanied by a critical assessment of the obtained dates. In this regard the suitability of C/N_{at} ratios along with $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ isotopic determinations was confirmed, similarly to previous studies. We also confirm that separation of purified component for dating should prevail over the will to destroy the minimum amount of the material.

ACKNOWLEDGMENTS

Funding provided in frame of National Science Centre (NCN) project “Old material with new methods: Using the latest bio-chemical analysis in studies of Mesolithic human remains from the Polish areas”, no. 2014/15/HS3/02184, the Ministry of Higher Education and Research grants from the Silesian University of Technology, Institute of Physics, no. BKS-104/RIF/2016, BKS-101/RIF/2017, BK-243/RIF/2016, and BK-229/RIF/2017.

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